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# Treatment of fish farm sludge supernatant by aerated filter beds and steel slag filters – Effect of organic loading rate

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## Abstract

The goal of our study was to develop an on-site cost-effective and extensive method for the treatment of supernatant of the sludge settling silo of fresh water fish farms. The main objectives of this study were 1) to determine the effect of the organic loading rate (OLR) on organic matter and nutrient removal efficiency of a pilot treatment system consisting of a series of aerated filter beds (AFBs) and electric arc furnace steel slag filters operated at different void hydraulic retention times ( $HRT_v$ ), 2) to validate the P-Hydrslag model as a design tool for slag filters and 3) to propose preliminary design options for fish farm sludge treatment. The 11.5 month experiment was divided into two phases: Phase 1 (P1) of 8.5 months with a low OLR to the AFBs of  $0.015 \text{ kg BOD}_5 \text{ m}^{-3} \text{ d}^{-1}$ , and Phase 2 (P2) of 3 months with a high OLR of  $0.5 \text{ kg BOD}_5 \text{ m}^{-3} \text{ d}^{-1}$ . The results showed that the OLR affected the organic matter mineralization and nitrification efficiency of AFBs. With a low OLR and an influent COD concentration of  $320 \text{ mg L}^{-1}$ , an average COD removal efficiency of 95% was observed while with a high OLR and an influent COD of  $5400 \text{ mg L}^{-1}$ , an average COD reduction of 65% was achieved. Due to the high OLR during P2, no nitrification took place probably due to the low

availability of oxygen in the AFBs. The TP removal efficiency of the AFBs was 50% during P1 (influent of 5.2 mg L<sup>-1</sup>), and 88% during P2 (influent of 110 mg L<sup>-1</sup>). All SCs showed a high o-PO<sub>4</sub> removal efficiency of >98% during P1 (average influent of 1.8 mg P L<sup>-1</sup>, effluent of 0.04 mg P L<sup>-1</sup>) and >85% during P2 (average influent of 8.7 mg P L<sup>-1</sup>, effluent of 0.65 mg P L<sup>-1</sup>) with a decrease in efficiency only observed over time in the SC that received the highest organic loading rate. It was concluded that with optimal loading rates, this compact biological and physicochemical semi-extensive treatment system offers a promising alternative to the high energy demand and maintenance treatment systems for organic matter and phosphorus removal, and that this treatment system could be applicable to other agro-environmental, municipal or residential effluents.

**Keywords:** aeration, calcium-rich, phosphorus, precipitation, reactive filter material, steel slag

## 1. Introduction

Fresh water trout farms discharge a significant amount of polluting nutrients, estimated to be for ammonium between 100-150 g N d<sup>-1</sup> per ton of annual fish production and for orthophosphate (o-PO<sub>4</sub>) between 20-60 g P d<sup>-1</sup> per ton (Boaventura et al., 1997). The main source of P in trout farms is raw sludge that is composed of fish excreta, uneaten food and fish carcass debris, and contains 30–84% of the total P discharged from fish farms (Lefrançois et al., 2010). Fish farms in Quebec, Canada are required to limit their global P discharge to the environment to 4.2 kg P per ton of fish produced, according to the Sustainable Development Strategy for Freshwater Aquaculture in Quebec (i.e. STRADDAQ). The commonly used approach to reduce pollutants from the effluent of

52 fish farms is by separating the solids from water through physical settling (Cripps and  
53 Bergheim, 2000). Once collected and settled, however, fish sludge still presents  
54 environmental problems mainly due to the management of the nutrient-rich sludge  
55 supernatant. Due to their remote location and relatively small volume of sludge  
56 supernatant (e.g. from 10 to 250 m<sup>3</sup> d<sup>-1</sup> in the fish farm of this study) the use of  
57 conventional intensive treatment systems is not commonly used. Therefore, more  
58 ecological, economically beneficial and low maintenance treatment options have to be  
59 found with the main goal of reducing P discharge.

60 Several studies (Comeau et al., 2001; Drizo et al., 2006; Chazarenc et al., 2010; Pratt  
61 and Shilton, 2010; Puigagut et al., 2011) were conducted to find the best methods for  
62 organic matter and nutrient (especially P) removal from fish farm wastewater. One  
63 ecological option is to use treatment wetlands (TWs) with filter systems as has been  
64 done for various types of wastewater from mainly domestic but also industrial, mining,  
65 agricultural, landfill leachate origins (Kadlec and Wallace, 2009). Common TW  
66 technologies, however, require large land area that is not always available. Furthermore,  
67 there are several constraints that derive from using TWs in cold climate conditions. One  
68 key element for efficient organic matter and nitrogen removal in TWs is the supply of  
69 oxygen that is needed for aerobic microbial processes. To address this issue, several  
70 studies have been done with intensified (i.e. engineered) TWs that use forced aeration  
71 (Muñoz et al., 2006; Nivala et al., 2013; Ouellet-Plamondon et al., 2006; Vymazal,  
72 2011).

73 In TWs, phosphorus is mainly precipitated in or sorbed onto filter media (Kadlec and  
74 Wallace, 2009). A sustainable solution could be to use reactive filter units containing  
75 replaceable material with a high P binding capacity (Brix et al., 2001; Drizo et al., 2006;  
76 Kõiv et al., 2010; Shilton et al., 2006). High P removal efficiency has been shown

77 through calcium (Ca)-phosphate precipitation using reactive filtration in Ca-rich  
78 alkaline filter materials (Liira et al., 2009; Claveau-Mallet et al., 2012, 2014). Potential  
79 materials for P precipitation include high Ca content industrial by-products such as  
80 metallurgical slags and ashes, in which Ca occurs in CaO form (lime) and/or Ca-  
81 silicates (Vohla et al., 2011).

82 As determined by the toxicity characteristic leaching procedure TCLP (U.S.  
83 Environmental Protection Agency, 1992) only non-hazardous filter materials should be  
84 selected for wastewater applications. Slags from 58 mills in the U.S. were tested and it  
85 was shown that although the total concentration of some metals in slag may be elevated,  
86 they remained tightly bound to the slag matrix and were often not readily leachable  
87 (Proctor et al., 2000). In current study electric arc furnace steel slag from Quebec,  
88 Canada was used and it has been considered as safe material according to TCLP  
89 procedure.

90 With reactive filter materials, appropriate biological pre-treatment for removing solids,  
91 organic matter and nutrients is crucial to provide a long lifetime of the reactive media  
92 by decreasing the risk of clogging and permitting the use of finer reactive filter media  
93 with higher P sorption capacity (Hedström, 2006). Phosphorus sorption sites may be  
94 blocked by organic matter or sorption may be reduced by competitive sorption of  
95 organic anions or by metal complexation (Nilsson et al., 2013). Supersaturation of pore  
96 water with respect to Ca and  $\text{o-PO}_4$  is essential for the precipitation of stable Ca-  
97 phosphate phases (House et al., 1999; Liira et al., 2009).

98 The goal of our study was to develop an on-site compact, cost-effective and  
99 environmentally friendly method for the treatment of the supernatant of fish farm sludge  
100 settling silos. The main objectives of this study : a) to determine the organic matter and  
101 nutrients removal efficiency of a pilot treatment system consisting of a series of aerated

102 gravel filter beds (AFBs, as a replacement for an aerated TW) and electric arc furnace  
 103 steel slag filters; b) to determine the effect of loading rate on performance; c) to  
 104 determine the effect of void hydraulic retention time ( $HRT_v$ ) on  $o\text{-PO}_4$  removal in slag  
 105 filters; d) to validate the P-Hydroslag model as a design tool for slag filters; and e) to  
 106 propose preliminary design options for fish farms.

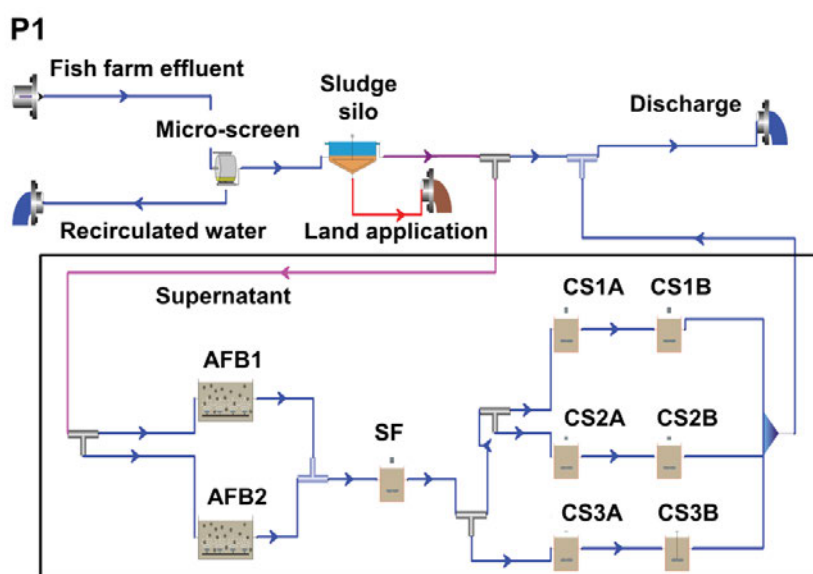
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## 108 2. Material and Methods

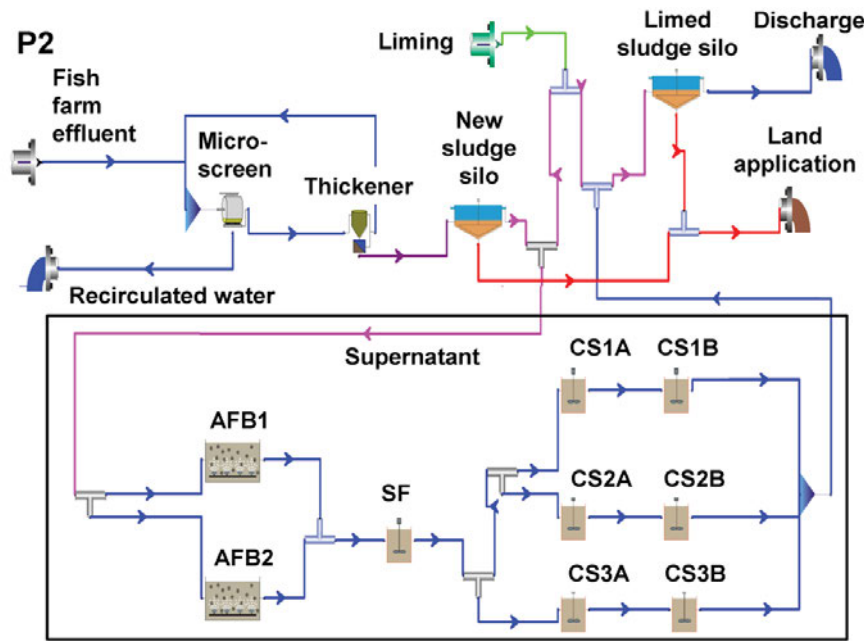
### 109 2.1. Site description

110 An on-site pilot experiment (Fig. 1) for the treatment of the supernatant from sludge  
 111 settling silo (i.e. reservoir) was established in 2010 at the “Ferme Piscicole des  
 112 Bobines”, a fresh water trout farm in East Hereford, Quebec, Canada that ran for a total  
 113 of 11.5 months. In the Bobines fish farm, there is a total 40 interior fish tanks. The  
 114 water used ( $10\,000$  to  $20\,400\text{ m}^3\text{ d}^{-1}$ ) originates from fresh groundwater (15%) and from  
 115 microscreened recirculated water (85%).

116



117



**Figure 1.** Schematic diagram of the fish farm treatment system and experimental system during Phase 1 (P1) and Phase 2 (P2) when the sludge supernatant contained a low (P1) or a high pollutant (P2) concentration. Abbreviations: AFB1 and AFB2 – two parallel aerated filter beds; SF – sacrificial slag filter; SC1A+SC1B, SC2A+SC2B, SC3A+SC3B – three parallel dual-stage steel slag columns with different void hydraulic retention times.

In 2010 and 2011 (during P1), about  $250 \text{ m}^3 \text{ d}^{-1}$  of screenings were collected and stored in a settling silo of  $250 \text{ m}^3$  with an HRT of about one day (Fig. 1). During the winter and spring of 2012 (just before P2), a sludge thickener, a new settling tank and a liming system for P removal were added. This new installation reduced the flowrate of concentrated sludge from  $250 \text{ m}^3 \text{ d}^{-1}$  to about  $10 \text{ m}^3 \text{ d}^{-1}$  and increased its pollutant concentration. The supernatant of the new sludge thickener ( $\sim 240 \text{ m}^3 \text{ d}^{-1}$ ) was returned to the microscreen and the thickened sludge ( $\sim 10 \text{ m}^3 \text{ d}^{-1}$ ) was sent to a newly built sludge silo with a volume of  $370 \text{ m}^3$  providing an HRT of 37 days. After settling in the new sludge silo, the highly concentrated supernatant was limed for P removal and then

135 stored in silo for limed sludge. Two times per year the thickened sludge from the new  
 136 sludge silo and the limed sludge were spread on agricultural land for fertilization and  
 137 land amendment (Fig. 1).

138

## 139 2.2. Experimental design

140 The experimental treatment system was composed of two parallel downflow saturated  
 141 aerated filter beds (AFBs) followed by a sacrificial slag filter (SF) and three parallel  
 142 dual-stage steel slag columns (SCs; Fig. 1). An insulated truck trailer was used to install  
 143 the experimental system to avoid freezing during winter periods.

144 The main design parameters of the experimental filter units are summarized in Table 1.

145

146 **Table 1.** Summary of design parameters of the experimental filter units in Phase 1 (P1)  
 147 and Phase 2 (P2). Abbreviations: AFB1 and AFB2 – two parallel aerated filter beds; SF  
 148 – sacrificial slag filter; SC1, SC2, SC3 – three parallel dual-stage steel slag columns.

Design parameters	Units	AFB1, AFB2	SF	SC1, SC2, SC3
Water flow conditions		saturated	saturated	saturated
		downflow	upflow	upflow
Filter size				
(diameter × height	m×m or	*1.0×1.0×1.0	0.45×0.8	0.3×1.3
or *length × width × height)	*m×m×m	(each AFB)		(each stage)
Water level in filter units	m	1.0	0.8	1.3
Volume of filter material	m <sup>3</sup> filter <sup>-1</sup>	0.90	0.13	*0.095
	(*or stage <sup>-1</sup> )			
Filter material		gravel	EAF steel slag	EAF steel slag



Design parameters	Units	AFB1, AFB2	SF	SC1, SC2, SC3
Particle size of material	mm	10-25	P1 SF1 = 20-40	
			P2 SF2 = 10-30	5-10
			P2 SF3 = 10-30	
Density of material	kg L <sup>-1</sup>	2.6	3.6	3.6
Porosity of filter (estimated)	%	38	45	40
Initial void volume	m <sup>3</sup>	0.36	0.052	0.038
Void hydraulic retention time (HRT <sub>v</sub> )	h	P1 = 48	P1 = 3.5	P1: 20, 12, 4.5
		P2 = 65	P2 = 4.8	P2: 30, 15, 6.0 (per stage)
Organic loading rate (OLR)	kg BOD <sub>5</sub> m <sup>-3</sup> d <sup>-1</sup>	P1 = 0.015	–	–
		P2 = 0.50		
Air flow direction		counter-	–	–
		current		

149

150 Even though the aerated filter beds (AFBs) are aerated saturated downflow biofilters,  
151 the lower design values of trickling filters (Metcalf and Eddy et al., 2014) were used to  
152 minimize maintenance of the AFBs. The design criterion chosen was based on a specific  
153 nitrogen removal rate per rock surface area (see also Table 3). The AFB effective  
154 volume was 0.36 m<sup>3</sup> each and they were periodically fed with sludge supernatant. The  
155 aeration system consisted of a porous diffuser that was connected to an air pump with a  
156 total air flow rate of 0.15 m<sup>3</sup> min<sup>-1</sup>. The supernatant treated in both AFBs was combined  
157 and then gravity flowed to the airtight upflow sacrificial filter with coarse steel slag (SF;  
158 Fig. 1). The main functions of the SF were to capture some inorganic carbon and P to  
159 improve the efficiency of the subsequent SCs. During P1, the coarse steel slag in SF  
160 was changed after 184 days of utilization to smaller size slag material (see Table 1). For  
161 P2, a new and finer steel slag was used (Table 1). For the SF, three periods are used,

162 SF1 (first 184 d) and SF2 (lasting 77 d) both during P1, and SF3 (lasting 77 d) during  
163 P2 (Table 1).

164 The effluent from the SF was pumped to three parallel dual-stage upflow EAF steel slag  
165 columns (SC1A+SC1B; SC2A+SC2B; SC3A+SC3B; Fig. 1) which main function was  
166  $\text{o-PO}_4$  precipitation. The dual-stage SCs had different  $\text{HRT}_V$  (Table 1) to determine the  
167 effect of hydraulic and pollutant loading rates on the efficiency of the EAF filters. All  
168 steel slag units were closed airtight to prevent atmospheric  $\text{CO}_2$  dissolution into the high  
169 pH water that would result in bicarbonate formation and calcium carbonate  
170 precipitation. Gravity flow was favored in the slag columns by installing a venting  
171 system connected to a siphon filled with mineral oil to minimize atmospheric  $\text{CO}_2$   
172 dissolution while allowing liquid flow.

173 The onsite pilot experiment was divided into two phases. Phase 1 (P1) lasted from the  
174 end of November 2010 to the middle of August 2011 for a duration of 8.5 months.  
175 During P1, the sludge supernatant was relatively diluted and the OLR of  $0.015 \text{ kg BOD}_5$   
176  $\text{m}^{-3} \text{ d}^{-1}$  to the experimental system was low. Phase 2 (P2) lasted from the end of May  
177 2012 to the end of August for total duration of 3.0 months. During P2 the sludge  
178 supernatant was highly concentrated and the OLR was high ( $0.5 \text{ kg BOD}_5 \text{ m}^{-3} \text{ d}^{-1}$ ).  
179 Between the two phases, during rebuilding of the fish farm sludge treatment system, the  
180 experimental system was at rest for 9 months with the filter units drained and all slag  
181 filters kept airtight.

182

### 183 2.3. Filter materials

184 Electric arc furnace (EAF) steel slag, a by-product of the steel industry, was used in the  
185 experimental filters and was produced by Arcelor Mittal of Contrecoeur, Quebec,  
186 Canada and obtained from “Minéraux Harsco”, also of Contrecoeur. The tested steel

slag has been used also in previous studies (Claveau-Mallet et al., 2012, 2014). Washed gravel (schist) used as a filter media in our aerated filter beds and as a bottom drainage layer in slag filters was obtained from a nearby quarry in East-Hereford, Quebec, Canada. The chemical composition of the EAF steel slag and gravel used in our experiment is presented as supplementary material in Supplementary Table S1.

## 2.4. Sampling and analytical methods

Samples from the influent and effluent of all filter units and from combined effluent of aerated filter beds (AFB<sub>cb</sub>) were taken once a week during the whole period of the operation using standard procedures (APHA et al., 2012). Extra samples were taken from the middle of the first slag columns (SC1A<sub>mid</sub>; SC2A<sub>mid</sub>; SC3A<sub>mid</sub>). The chemical oxygen demand (COD), carbonaceous biochemical oxygen demand (CBOD<sub>5</sub>), total suspended solids, (TSS), volatile suspended solids (VSS), total Kjeldahl nitrogen (TKN), ammonium (NH<sub>4</sub>), nitrate plus nitrite (NO<sub>x</sub>), total phosphorus (TP), orthophosphate (o-PO<sub>4</sub>), calcium (Ca<sup>2+</sup>), alkalinity and pH were determined according to Standard Methods (APHA et al., 2012). The Statistica 7.0 software was used for data analyses for which a level of significance of  $\alpha=0.05$  was used in all cases. In the “Results and discussion” section, significant differences were outlined when  $p < 0.05$  was obtained.

## 2.5. Numerical simulations

Slag column operation was simulated using the P-Hydroslag model written in the PHREEQC software, using slag exhaustion equations that were determined previously for the “Minéraux Harsco” EAF slag (Claveau-Mallet et al. 2014). The exhaustion

equations were adapted to consider 40% porosity, resulting in the following equations (1) and (2):

$$\log(k_{diss}) = -0.3688B - 5.46 \quad (1)$$

$$pH_{sat} = 1.71B^2 - 3.835B + 12.44 \quad (2)$$

where  $k_{diss}$  is the slag dissolution kinetic constant ( $M\ Ca\ s^{-1}$ ),  $pH_{sat}$  is the slag saturation pH and  $B$  is the total leached CaO in the slag filter ( $mol\ L^{-1}$ ).

Slag column influent solutions were reproduced using chemicals ( $CaCl_2$ ,  $KH_2PO_4$ ,  $K_2HPO_4$ ,  $CaO$ ,  $NH_4Cl$ ,  $NaHCO_3$  and  $KCl$ ) in the REACTION datablock. Solutions were equilibrated with hydroxyapatite, monetite and calcite in the EQUILIBRIUM\_PHASES datablock. Resulting solutions are shown in Table 2. Simulations were conducted for both phases P1 and P2, and for the first columns SC1A, SC2A and SC3A. Simulated and experimental results were compared. Additional simulations using the AFB effluent as column influent were conducted to assess the effect of removing the SF.

**Table 2.** Composition of simulated influent solutions of the slag columns (AFBs effluent feeding the SF and SF effluent feeding the SCs).

Phase	pH	$Ca^{2+}$	TIC	o- $PO_4$	$NH_4$	Alkalinity
	-	$mg\ L^{-1}$	$mg\ C\ L^{-1}$	$mg\ P\ L^{-1}$	$mg\ N\ L^{-1}$	$mg\ CaCO_3\ L^{-1}$
AFB P1	7.19	24.6	13.4	2.42	-	51.6
SF P1	8.27	28.1	13.4	2.42	-	60.3
AFB P2	7.39	23.4	159.8	13.6	210	645.3
SF P2	8.07	30.7	145.2	13.6	210	663.4

231

### 232 **3. Results and discussion**

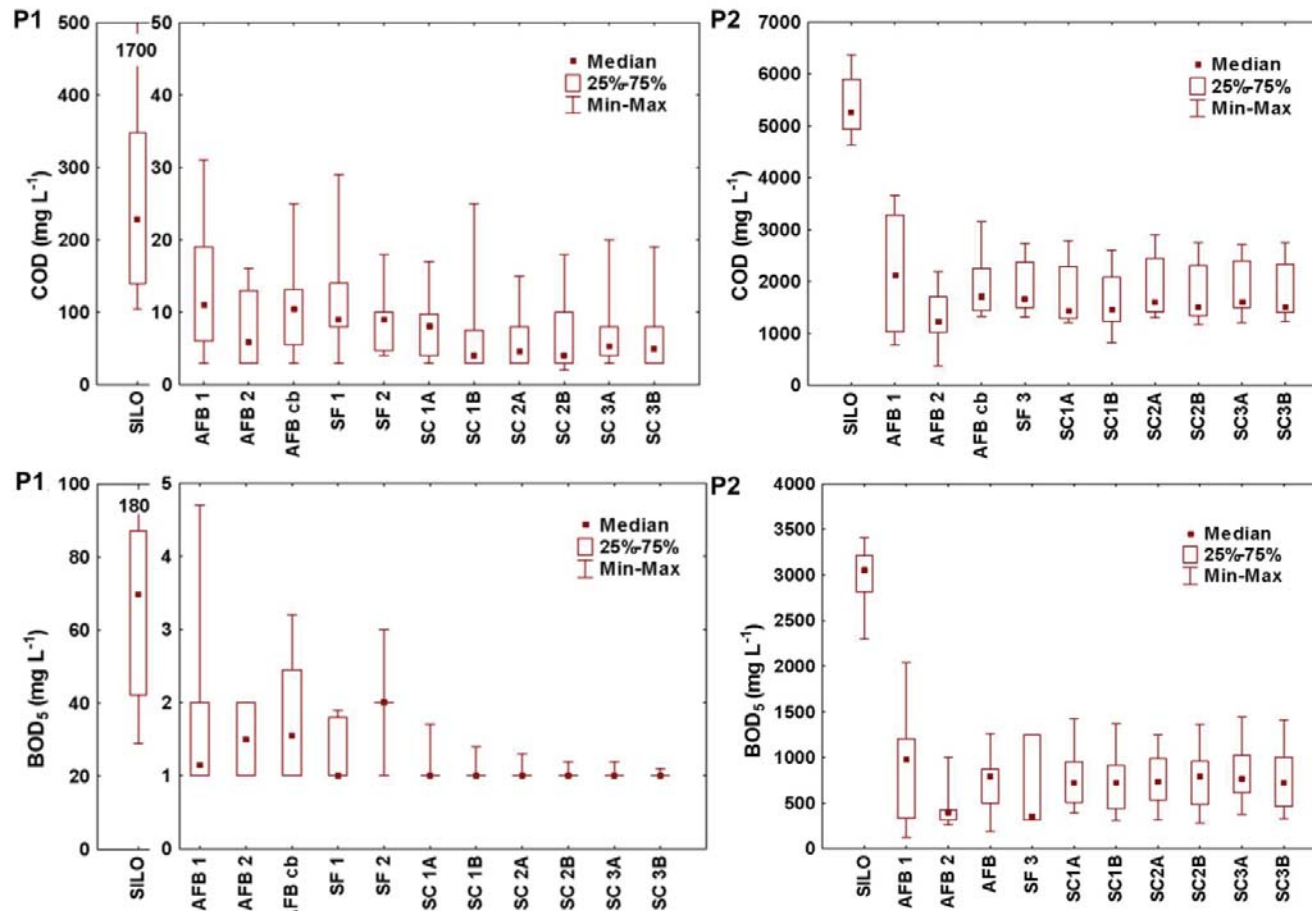
233 During P1, the supernatant from the sludge silo had a composition similar to typical low  
234 strength municipal wastewater while it was much more concentrated during P2  
235 (Supplementary Table S2). Standard deviation (SD) values indicate that during both  
236 phases the influent concentration of most pollutants was quite variable over time  
237 (Supplementary Table S2). The variability was due to the varying amount of solids in  
238 the sludge silo with peak concentrations due to the silo being full of solids and in need  
239 of cleaning.

240

#### 241 3.1. Removal of organic matter, solids and nitrogen

242 The main functions of the aerated filter beds were to remove solids and to oxidize  
243 organic matter and ammonium. During P1, the aerated filter beds (AFB<sub>cb</sub> – shown as  
244 combined effluent) were very efficient in mineralizing organic matter and removing  
245 solids (Supplementary Table S2; Fig. 2). Average reductions obtained in the AFBs were  
246 95.3% for COD, 97.3% for BOD<sub>5</sub>, 95.8% for TSS and 94.9% for VSS resulting in  
247 effluent values of less than 3 mg TSS L<sup>-1</sup> (Fig. 2). During P2, the degradation of organic  
248 matter in AFBs was quite good (Supplementary Table S2; Fig. 2), with an average  
249 removal of 65% for COD and 71% for BOD<sub>5</sub> (Fig. 2). However, the AFB effluent COD  
250 and BOD<sub>5</sub> values remained high at 1730 and 895 mg O<sub>2</sub> L<sup>-1</sup>, respectively.

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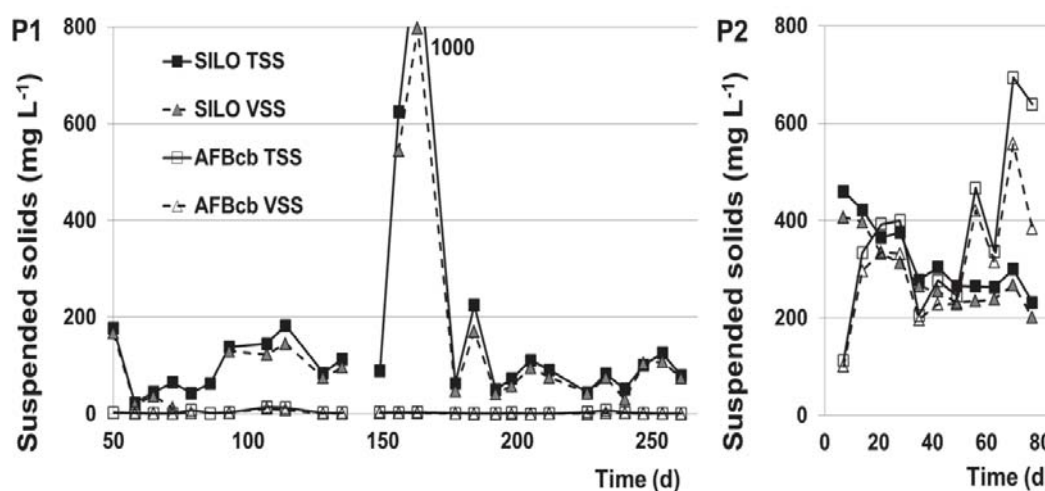


252  
 253 **Figure 2.** Organic matter removal by the various treatment units (presented as changes in BOD<sub>5</sub> and COD values) during Phases 1 and 2 (P1 and  
 254 P2) of the experiment. Note: The two AFBs and the three sets of two columns in series of SCs were operated in parallel.

255

256 A rapid increase in TSS and VSS concentration from the sludge silo was observed after  
257 about 170 days of operation during P1 (Fig. 3) indicating that the sludge silo needed to  
258 be emptied. During P2, the effluent from the AFBs had a higher TSS and VSS  
259 concentration than the influent, indicating that the AFBs should have been backwashed  
260 to prevent excessive solids accumulation. Backwashing could be achieved by sending  
261 AFB treated water and air at the bottom of the AFB to clean the bed of excess biosolids,  
262 as is typically done for biofilters (Metcalf and Eddy et al., 2014). The recovered solids  
263 would then be sent to a settling tank for treatment. With low loaded AFBs, such an  
264 operation may have to be done once a month or less.

265



266

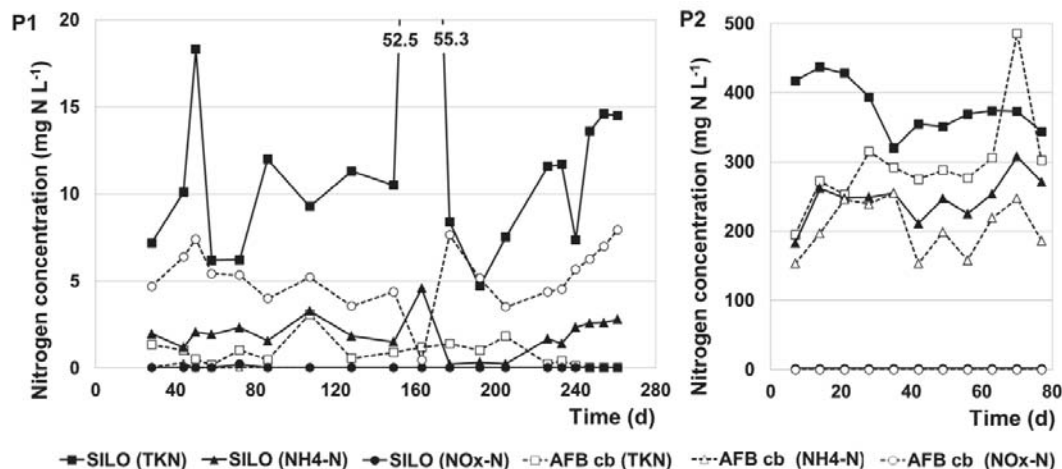
267 **Figure 3.** Evolution of total suspended solids (TSS) and volatile suspended solids  
268 (VSS) concentration in the combined effluents of the aerated filter beds (AFB<sub>cb</sub>)  
269 effluent during P1 and P2.

270

271 Efficient ammonification of organic nitrogen (TKN minus NH<sub>4</sub>) and nitrification were  
272 observed in the AFBs during P1 (average effluent TKN only 0.9 mg N L<sup>-1</sup>) as confirmed

273 by the increase of oxidized nitrogen concentration in the effluent (from 0.05 to 5.1 mg  
 274 L<sup>-1</sup>, Fig. 4).

275



276

277 **Figure 4.** Nitrogen removal and transformation (presented as changes in TKN, NH<sub>4</sub>-N  
 278 and NO<sub>x</sub>-N concentrations) in the combined effluent of the aerated filter beds (AFB<sub>cb</sub>)  
 279 during Phase 1 and 2 (P1; P2).

280

281 During P2, the overall efficiency of TKN removal by the system was 32% of which  
 282 20%, on average, was achieved by the AFBs (p>0.03; Fig. 4). The AFBs removed only  
 283 17% of ammonium and there was no increase in NO<sub>x</sub> concentration. This can be  
 284 explained by the BOD<sub>5</sub> concentration that was too high to allow efficient nitrification  
 285 (Metcalf and Eddy et al., 2014). In summary, the AFB is suitable technology for  
 286 sufficient pre-treatment of fish farm supernatant when appropriate loading rates are used  
 287 (shown in details in Section 3.4).

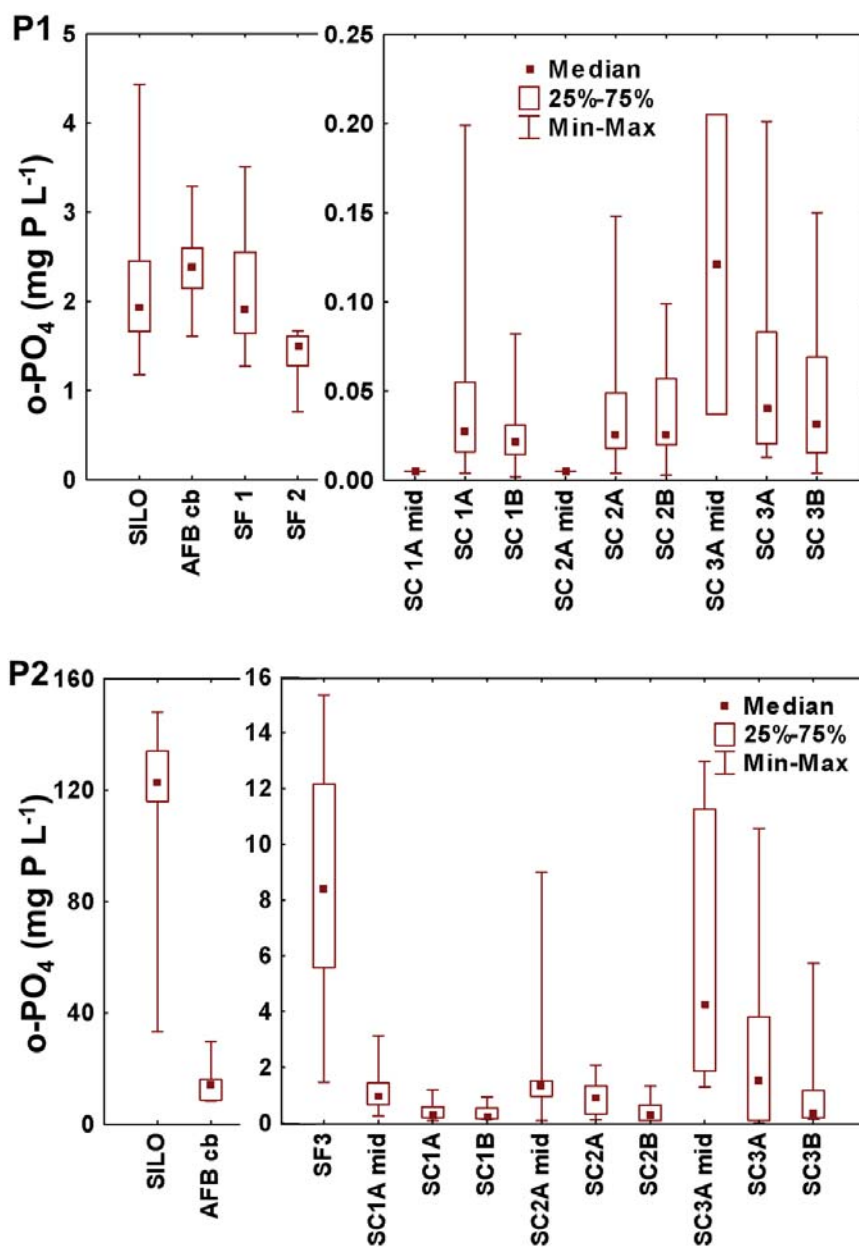
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### 289 3.2. Removal of total phosphorus and orthophosphate



290 The TP removal in the AFBs during P1 was on average 35% (effluent 2.6 mg TP L<sup>-1</sup>)  
 291 while the o-PO<sub>4</sub> concentration increased by about 10% due to hydrolysis (effluent  
 292 concentration 2.4 mg L<sup>-1</sup>).

293

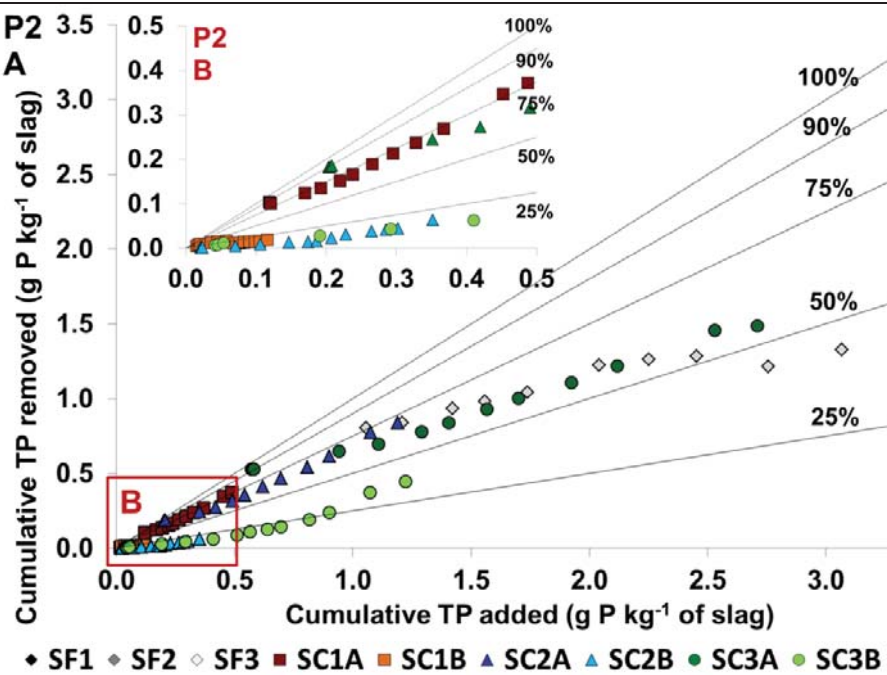
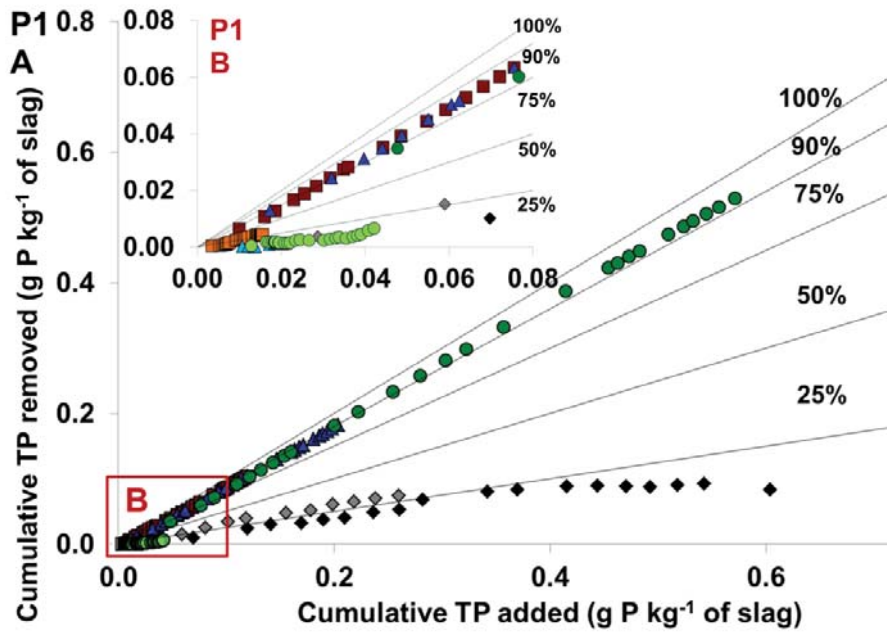


295

296 **Figure 5.** Evolution of orthophosphate (o-PO<sub>4</sub>) during P1 and P2 in the filter units.

297

298 During P2, an average 78% of TP and 86% of o-PO<sub>4</sub> was removed in the AFBs (Fig. 5).  
299 This high efficiency was unexpected and could be attributed to phosphate precipitation  
300 with calcium, iron or aluminium ions present in the sludge supernatant. In P2, sludge  
301 supernatant had a sufficiently high calcium concentration (145 mg L<sup>-1</sup> soluble) to induce  
302 precipitation. Calcium leaching of the AFBs was also observed in P1.  
303  
304 During P1, the sacrificial slag filter showed an average o-PO<sub>4</sub> removal of only 17% and  
305 34% in SF1 and SF2, respectively (Fig. 5). During P2, the cumulative amount of TP  
306 removed and the removal efficiency in SF3 was higher than in SF1 and SF2 during P1  
307 (Fig. 6), with an average o-PO<sub>4</sub> removal of 47%. During P2, the efficiency of SF3 was  
308 comparable with SC3A – the slag column that received the highest loading rates during  
309 the whole experiment. Similarly to the constant decrease in the TP removal efficiency  
310 there was concomitant decrease in the pH value of the effluent from pH 9.0 to pH 8.0.  
311 Thus, the SF increased somewhat the pH of the treated supernatant, but contributed only  
312 slightly to phosphorus removal.  
313



**Figure 6.** Ratio between cumulative total phosphorus (TP) added and removed in steel slag filters (SF and SCs) during Phase 1 and 2 (P1 and P2). All SCs used during P1 continued to be used during P2, which explains why the first data points at the beginning of P2 (graph B) are not located at the origin of the graph.

321 *3.2.1. Phosphorus removal in dual-stage steel slag columns*

322 All dual-stage slag columns (SCs) removed on average more than 96% of TP during P1  
323 with a stable effluent concentration averaging from 0.10 to 0.17 mg P L<sup>-1</sup>. During P2, a  
324 lower TP removal efficiency of 85-91% was observed. This resulted in an effluent TP of  
325 5.4 to 7.4 mg L<sup>-1</sup> but a relatively low o-PO<sub>4</sub> concentration of 0.3 to 1.1 mg P L<sup>-1</sup>  
326 (Supplementary Table S2; Fig. 5). Slag columns removed o-PO<sub>4</sub> by Ca-phosphate  
327 precipitation (Claveau-Mallet et al., 2012) and particulate P mainly by physical  
328 filtration. Particulate P removal could be increased by increasing the HRT<sub>V</sub> as this  
329 would slow down the water velocity inside the filter material and increase the solids  
330 filtration (Claveau-Mallet et al., 2012). As particulate matter can clog slag columns,  
331 sufficient pre-treatment is needed to prevent solids accumulation (Hedström, 2006).  
332 During P1, almost all o-PO<sub>4</sub> was precipitated in the first stage of the SCs (e.g. in SC1A  
333 >97%), and the final effluent o-PO<sub>4</sub> concentration varied from 0.03 to 0.05 mg P L<sup>-1</sup>  
334 (Fig. 4). There were only slight differences in the final TP and o-PO<sub>4</sub> concentration in  
335 between the SCs during P1 even though there was a tendency for the columns with  
336 longer HRT<sub>V</sub> to remove phosphate a little more efficiently (Fig. 5). The SC3A+SC3B  
337 with the shortest HRT<sub>V</sub> (4.5 h + 4.5 h) and highest P loading rate had the highest  
338 orthophosphate removal efficiency during P1 (96%). Furthermore, the SC3A started to  
339 show its first signs of saturation during the last month of operation when the effluent o-  
340 PO<sub>4</sub> concentration increased rapidly from 0.03 to as high as 10.6 mg P L<sup>-1</sup> (see also Fig.  
341 8). In the downstream SC3B the average o-PO<sub>4</sub> removal efficiency during P2 remained  
342 above 90% (TP removal 63%), but during last two weeks of operation, the o-PO<sub>4</sub>  
343 removal efficiency of this column decreased to 58%.

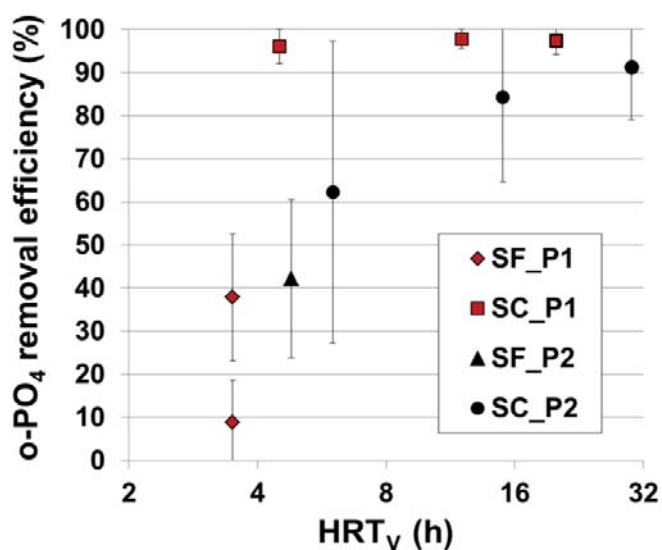
344

345 During P2, the effect of cumulative loading on the SCs efficiency was observed (Fig. 6).  
 346 Over time, the SC3A+SC3B with higher TP loading removed less TP. During this  
 347 phase, SC1A+SC1B removed up to 0.4 g P kg<sup>-1</sup> and SC2A+SC2B a total of 0.9 g P kg<sup>-1</sup>  
 348 of slag. The first column SC3A of dual-stage slag filter which received the highest TP  
 349 loading, removed a total of 1.4 g P kg<sup>-1</sup> slag.

350

351 The effect of HRT<sub>V</sub> on the average o-PO<sub>4</sub> removal efficiency in slag filters is shown on  
 352 Figure 7. The SCs with longer HRT<sub>V</sub> had high and relatively stable P removal  
 353 efficiency during both phases, and an effluent o-PO<sub>4</sub> concentration over 2 mg L<sup>-1</sup> in  
 354 SC2A was only observed during the last week of operation. The only slag column that  
 355 showed significantly lower o-PO<sub>4</sub> removal efficiency during P2 was SC3A. These  
 356 results indicate that there is a clear effect of HRT<sub>V</sub> on o-PO<sub>4</sub> removal especially when a  
 357 high OLR is applied to the system. At a high OLR, the slag filters should be operated at  
 358 a minimum HRT<sub>V</sub> of 30 hours to ensure efficient o-PO<sub>4</sub> removal.

359



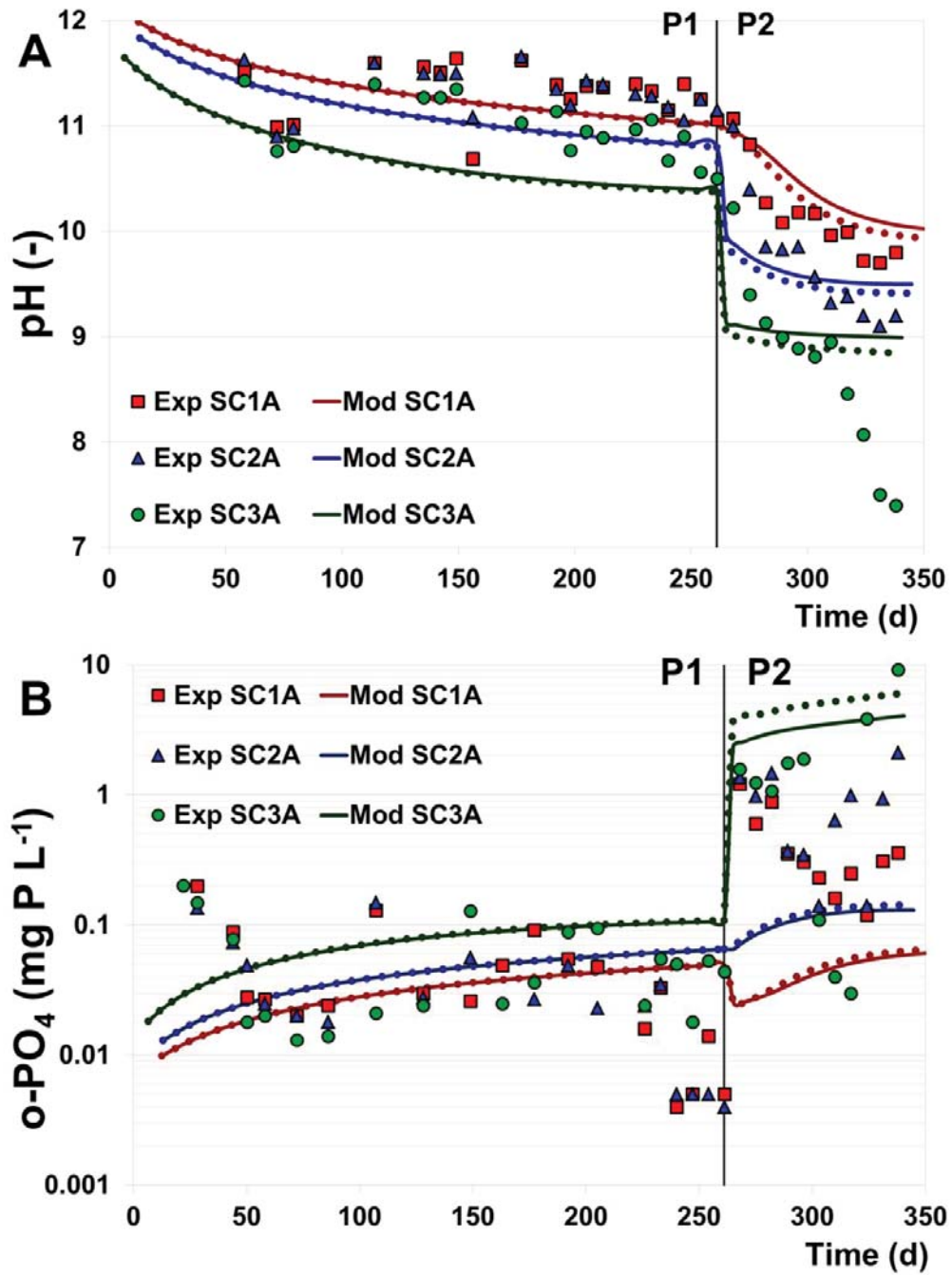
360

361 **Figure 7.** Effect of void hydraulic retention time (HRT<sub>V</sub>) on average orthophosphate (o-  
 362 PO<sub>4</sub>) removal efficiency during P1 (red; low o-PO<sub>4</sub> load) and P2 (black; high o-PO<sub>4</sub>

load) in sacrificial slag filters (SFs) and dual-stage slag columns (SCs). Error bars denote standard deviations.

The evolution of SCs effluents composition is compared to P-Hydroslag model simulation results on Figure 8. The experimental curves of pH were reasonably well reproduced by simulation, except for SC3A at the end of P2, where the observed pH drop was larger than that simulated. The o-PO<sub>4</sub> simulated curves generally reproduced experimental curves, but they were less accurate during P2. The HRT<sub>V</sub> effect predicted by the model was observed in experimental results. The model properly predicted an o-PO<sub>4</sub> concentration over 1.0 mg P L<sup>-1</sup> for SC3A during P2.

The model also predicted the effect of changing from P1 to P2, and showed that removing the SF did not significantly decrease the SCs performance (solid vs dotted lines on Fig. 8). This result constitutes the first calibration of the P-Hydroslag model with a field-scale application and validates the potential of this model as a design tool. The current version of the model is intended to simulate secondary effluent containing low suspended solids content, as it considers soluble influent only. The model still needs improvement concerning some model assumptions regarding the effect of particulate matter accumulation and the refinement of the methods to measure kinetic parameters. These aspects are included in a forthcoming version of the model (Claveau-Mallet et al., In preparation).



**Figure 8.** Simulation results of the P-Hydroslag model and comparison with experimental data for A) pH and B) o-PO<sub>4</sub>. Simulated curves with sacrificial slag filter (SF; solid lines) and without SF (dotted lines) are shown.

390 A longer  $HRT_V$  should improve the contact between the substrate and the wastewater  
391 and increase P removal. Liira et al. (2009) using hydrated oil shale ash, a material with  
392 similar removal mechanisms to slag, showed that at a long  $HRT_V$ , a dense and tightly  
393 packed layer of acicular calcite crystallites forms that gradually covers the entire surface  
394 of the particles. As a result, the dissolution of Ca is inhibited, and the phosphate  
395 precipitation decreases.

396 From experimental results and according to numerical simulations, it was confirmed  
397 that the  $HRT_V$  plays a central role in the long-term operation of slag filters. Previous  
398 research (Shilton et al., 2006; Vohla et al., 2011) showed that slag filters are capable to  
399 precipitate much higher amounts of  $o\text{-PO}_4$  than observed during this experiment (e.g. P  
400 retention capacity of EAF slag  $6.4 \text{ g P kg}^{-1}$  of slag; Claveau-Mallet et al., 2012). Such  
401 high capacity was achieved in batch and lab-scale experiments with a synthetic P  
402 containing feed and there are only few studies done with onsite and full-scale systems  
403 during extended periods of time. Shilton et al. (2006) presented results of full-scale  
404 treatment plant with reactive steel slag filters where an average TP removal efficiency  
405 of 77% and a maximum removal level of  $1.2 \text{ g TP kg}^{-1}$  (total of 20 tons of P removed)  
406 of slag were reported during the first 5 years of operation.

407 In full-scale systems, the  $HRT_V$  of the slag filter should be chosen to favour compact  
408 crystallization (crystal growth and not formation of new crystal seeds) of Ca-phosphate  
409 and to minimize the precipitation of Ca-carbonates (Claveau-Mallet et al., 2012). The  
410 minimum  $HRT_V$  required to support crystal growth and long-term operation is related to  
411 the hydroxyapatite crystal growth rate, which is related to the composition of the  
412 wastewater (Claveau-Mallet et al., 2012, 2014). Finding the optimal  $HRT_V$  according to  
413 the characteristics of wastewater is possible by using the P-Hydroslag model.

414



### 415 3.4. Preliminary design of the hybrid treatment system

416 Preliminary full-scale design options for the treatment of sludge settler supernatant  
417 based on the average composition of the supernatant from “Les Bobines” fish farm  
418 during P1 and P2, and experimental results of this study were proposed (see Table 3).

419 For low strength supernatant (P1), the conditions were characterized by a relatively high  
420 flowrate ( $50 \text{ m}^3 \text{ d}^{-1}$ ) but a low pollutant concentration that was similar to those of a  
421 typical low strength municipal wastewater. For high strength supernatant (P2), the  
422 conditions were for a lower flowrate ( $10 \text{ m}^3 \text{ d}^{-1}$ ) but a higher pollutant concentration.

423 A low-rate AFB (version P1 in Table 3) with nitrification was chosen for supernatant  
424 similar to the one used during P1, while a high-rate AFB with recirculation was chosen  
425 for wastewater similar to P2. Aeration was provided for  $\text{BOD}_5$  and ammonia oxidation  
426 for P1 and P2. AFB effluent recirculation was not tested during this experiment but such  
427 a mode of operation could improve the treatment efficiency when dealing with high  
428 strength wastewater.

429 A potential alternative for the AFBs when treating a low concentration supernatant  
430 could be more passive and low maintenance treatment wetlands (Kadlec and Wallace,  
431 2009). The sacrificial filter with coarse slag used in the experiment was not incorporated  
432 in this full-scale design because the SF increased somewhat the pH of the treated  
433 supernatant, but contributed only slightly to bicarbonate and phosphorus removal.  
434 Furthermore, the simulation results showed that removing the SF did not significantly  
435 decrease the SCs performance (Fig. 8).

436

437 **Table 3.** Preliminary full-scale design parameters for the treatment of low (P1) and high  
438 strength (P2) supernatant of a freshwater fish farm sludge settling tank, consisting of a)  
439 an aerated filter bed (AFB) and b) a reactive slag filter.

Wastewater type		P1 (low strength)	P2 (high strength)
<b>Aerated filter bed</b>			
<b>1) AFB influent data (see Supplementary Table S2)</b>			
<b>2) Objectives</b>			
TKN removal efficiency	%	90	10
BOD removal efficiency	%	80	50
<b>3) AFB design criteria (results from trickling filter design, Metcalf and Eddy et al., 2014)</b>			
Type of AFB		low-rate	high-rate (with recirculation)
Specific surface area in the reactor	$\text{m}^2 \text{m}^{-3}$	60	60
Hydraulic loading rate	$\text{m d}^{-1}$	3.6	1.04
Organic loading rate	$\text{kg BOD}_5 \text{m}^{-3} \text{d}^{-1}$	0.13	1.57
Specific TKN removal rate per rock area	$\text{g m}^{-2} \text{d}^{-1}$	0.40	0.33
<b>4) Design values</b>			
Filter volume (empty bed)	$\text{m}^3$	28	19
Filter depth	m	2	2
Filter area	$\text{m}^2$	13.9	9.6
Particle size of material	mm	10-20	10-20
Aeration rate	$\text{m}^3 \text{min}^{-1}$	0.83	2.92
<b>Slag filter</b>			
<b>1) Slag filter influent data (see Supplementary Table S2)</b>			
<b>2) Objectives</b>			
P removal efficiency	%	80	80
<b>3) Slag filter design criteria (results from numerical simulations using the P-Hydroslog model)</b>			
Void hydraulic retention time	h	8	30
Longevity	pore volumes	5400	315
	years	5	1
P retention capacity	$\text{g P kg}^{-1} \text{slag}$	4.5	1.7
<b>4) Design values</b>			
Filter volume (empty bed)	$\text{m}^3$	42	31
Filter depth	m	2	2
Filter area	$\text{m}^2$	21	15.5
Particle size of material	mm	5-10	5-10

440

441

442 The reactive slag filter design was based on numerical simulations performed with the  
443 P-Hydroslog model. Using the information from this experiment and previous research  
444 (e.g. Barca et al., 2012; Claveau-Mallet et al., 2013, 2012; Drizo et al., 2002; Liira et al.,  
445 2009; Shilton et al., 2006) it was estimated that during P1, the optimal  $\text{HRT}_V$  for slag

446 columns should be about 8 h (Table 3). For a more concentrated supernatant as during  
447 P2, a  $HRT_V$  of 30 h was proposed. The longevity and P retention capacity of slag filters  
448 were determined using numerical simulations as previously presented; considering that  
449 the filter longevity was reached when the effluent  $o\text{-PO}_4$  concentration was above 1 mg  
450 P  $L^{-1}$ . A safety factor of 2 was used for longevity. The resulting longevity was 5400  
451 pore volumes for P1 and much lower for P2 (315 pore volumes). Longevity expressed  
452 in pore volumes allows a more direct comparison of operating conditions of designs of  
453 P1 and P2, independently of the  $HRT_V$ . The longevity expressed in total time was 5  
454 years for P1 and 1 year for P2. In the latter case, the designer could choose between  
455 frequent replacement of the media (every 1 or 2 years) or increasing the size of the slag  
456 filter.

457 For best performance and for preventing short-circuiting in the filter, a vertical upflow  
458 feeding mode with a uniform influent distribution system at the bottom of the slag filter  
459 is proposed. The slag filters should be built deeper than usual filter units and as airtight  
460 as possible to allow water to flow but to minimize gaseous  $CO_2$  dissolution, thus  
461 reducing calcium carbonate precipitation and rapid pH lowering in the slag. At least two  
462 filters in series should be constructed to provide redundancy in the treatment system.

463 Bringing down the high effluent pH of the slag column is needed. At a fish farm, the  
464 slag filter effluent could simply be diluted in the main effluent by a ratio of at least  
465 100:1 prior to discharge to the receiving stream, bringing the final pH value well below  
466 9.5 which is the required pH limit in Quebec, Canada. If dilution into a larger stream is  
467 not an option, then it is possible to install a post treatment unit for pH adjustment. A  
468 peat filter installed downstream of an reactive hydrated ash filter was tested in lab-scale  
469 by Liira et al. (2009) for pH neutralization from  $> 10$  to pH 7-8. An alternative option is  
470 pH neutralization with gaseous  $CO_2$  (Sawyer et al., 2003).

471 Once the slag filter capacity is "exhausted", an extra TCLP test (U.S. Environmental  
472 Protection Agency, 1992) should be run to determine if saturated material should be  
473 disposed of as a hazardous waste, a municipal solid waste or can be valorised by land  
474 application as a soil amendment (Bird and Drizo, 2009). However, the direct use of non-  
475 soluble phosphate such as from hydroxyapatite would require an effective and  
476 economical means of solubilisation. This problem might be solved with the use of  
477 microorganisms (e.g. phosphate-solubilizing bacteria, Richardson, 2001) and  
478 phosphate-solubilizing fungi (Whitelaw, 1999). Other research demonstrated that plant  
479 root exudates produce organic acids that are strong enough to dissolve P even from  
480 hydroxyapatite and the P-saturated filter materials could be source of slow release P  
481 (Cucarella et al., 2007; Kõiv et al., 2012).

482 Highly concentrated influents would require a more intensive biological pre-treatment  
483 upstream of the reactive slag filters and the slag would saturate faster and would need to  
484 be changed at a higher frequency. Therefore, when considering that most of freshwater  
485 fish farms produce sludge supernatant that is more similar to the characteristics of P1 it  
486 was concluded that a hybrid treatment system consisting of an aerated filter bed or a  
487 treatment wetland followed by reactive slag filters would provide efficient pollutants  
488 removal from the supernatant.

489

## 490 **Conclusions**

491 The goal of our study was to develop an on-site compact, cost-effective and extensive  
492 system for the treatment of fish farm sludge supernatant. The conclusions of the project  
493 regarding initial objectives are the following:

- 494 a) The tested system composed of a downflow AFB followed by a SF and a series  
495 of two SCs achieved a mean effluent concentration (and mean % of removal) of

496 6.0 mg COD L<sup>-1</sup> (98%), 3.0 mg TSS L<sup>-1</sup> (98%), 0.5 mg TKN L<sup>-1</sup> (96%), 0.13 mg  
 497 NH<sub>4</sub>-N L<sup>-1</sup> (93%), 0.10 mg TP L<sup>-1</sup> (98%) and 0.05 mg o-PO<sub>4</sub>-P L<sup>-1</sup> (98%) in a  
 498 low concentration supernatant and a low OLR (0.015 kg BOD<sub>5</sub> m<sup>-3</sup> d<sup>-1</sup>; Phase 1);  
 499 and 1490 mg COD L<sup>-1</sup> (67%), 200 mg TSS L<sup>-1</sup> (37%), 256 mg TKN L<sup>-1</sup> (32%),  
 500 188 mg NH<sub>4</sub>-N L<sup>-1</sup> (24%), 5.9 mg TP L<sup>-1</sup> (96%) and 0.5 mg o-PO<sub>4</sub>-P L<sup>-1</sup> (99.5%)  
 501 in a high concentration supernatant and a high OLR (0.5 kg BOD<sub>5</sub> m<sup>-3</sup> d<sup>-1</sup>; Phase  
 502 2). The system was especially efficient in removing o-PO<sub>4</sub>, achieving effluent  
 503 concentration below 1.0 mg P L<sup>-1</sup> consistently during the whole experimental  
 504 period and according to P-Hydroslog model results could have continued to do  
 505 so for 5 years (when average o-PO<sub>4</sub> loading similar to P1).

506 b) The OLR had a substantial effect on the organic matter mineralization and  
 507 nitrification efficiency of aerated filter beds. At a low OLR, the AFBs were  
 508 efficient at removing COD (95%) and nitrifying the effluent while at a high  
 509 OLR, COD removal was reduced to 65% and no nitrification took place.

510 c) A high HRT<sub>V</sub> (>12 h) of SC resulted in higher o-PO<sub>4</sub> removal efficiency for both  
 511 phases compared to SC with HRT<sub>V</sub> < 6 h.

512 d) The P-Hydroslog model was used to predict the slag filter behaviour.  
 513 Experimental pH and o-PO<sub>4</sub> in the effluent were reasonably well reproduced by  
 514 simulated results, confirming the potential of the model as a design tool. The  
 515 general effect of HRT<sub>V</sub> and phase change was correctly predicted by the model.

516 e) Preliminary design options for a fish farm supernatant treatment system were  
 517 proposed for two types of supernatant (similar to Phases 1 or 2). The suggested  
 518 design included one AFB followed by a steel slag filter, without a sacrificial slag  
 519 filter. The design of the AFB was based on the OLR and the design of the slag  
 520 filter was based on numerical simulations with the P-Hydroslog model. The

521 proposed AFB  $HRT_V$  was 2.4 and 96 h for P1 and P2, respectively, while the  
522  $HRT_V$  of slag filter was 8 and 30 h for P1 and P2. The expected longevity was at  
523 least 20 years for the AFB (both phases), 5 years for the slag filter in P1, and 1  
524 year for the slag filter during P2.

525

526 It was concluded that with proper loading rates, this compact biological and physico-  
527 chemical treatment system offers a good alternative to the high energy demand and high  
528 maintenance treatment systems for organic matter and phosphorus removal, and that this  
529 treatment system could be applicable to other agro-environmental, municipal or  
530 residential effluents.

531

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540

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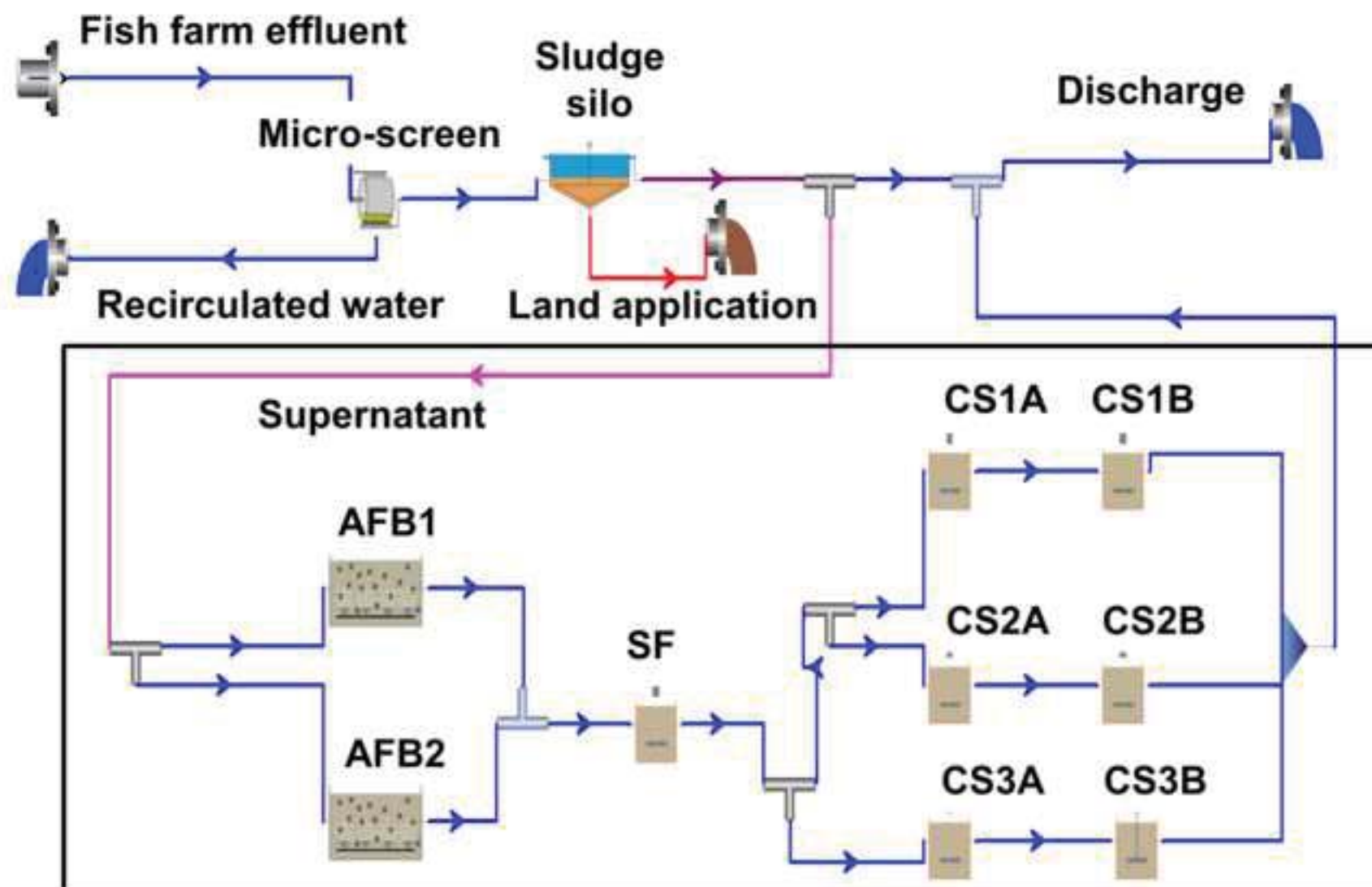
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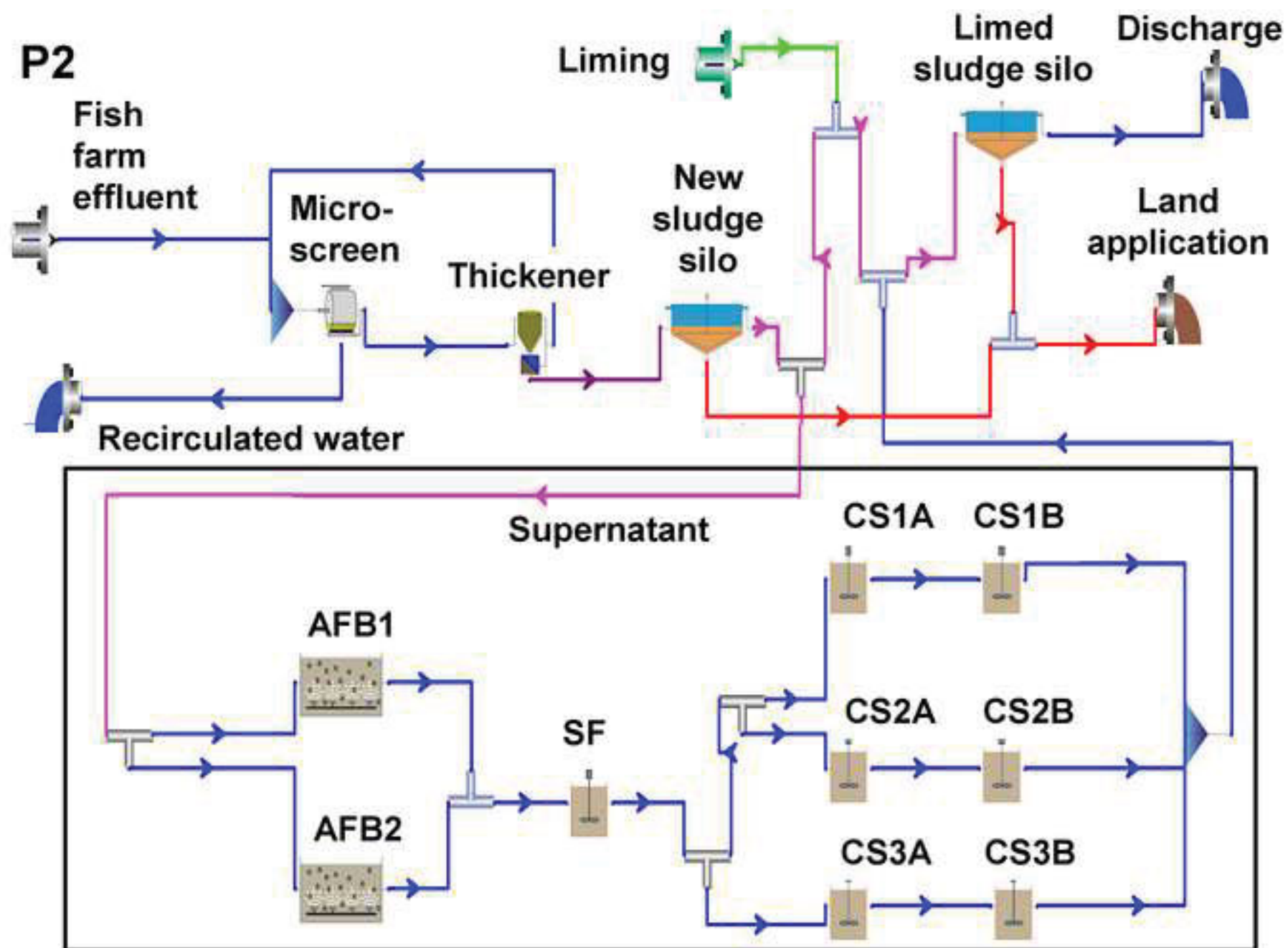
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**P1**



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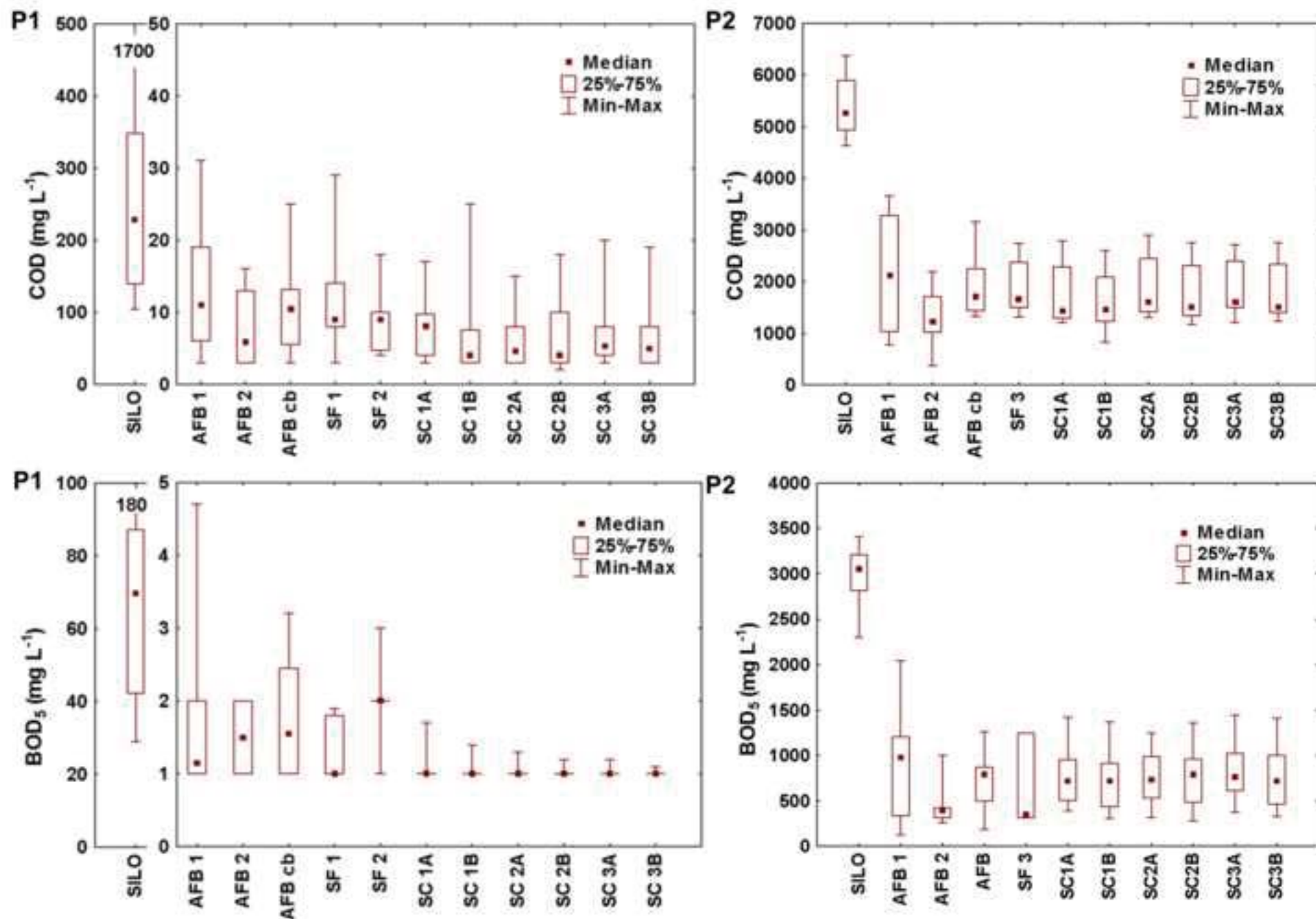
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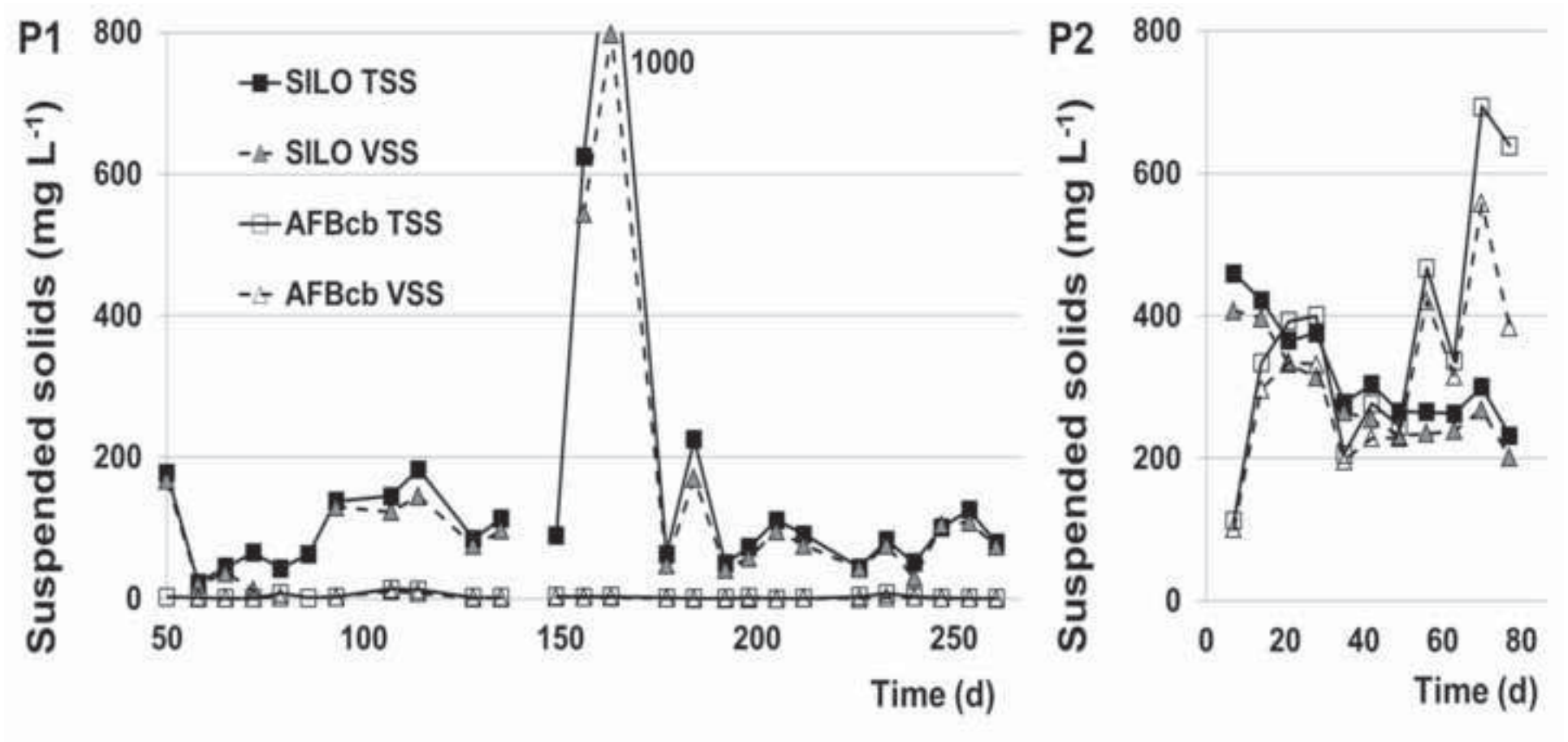


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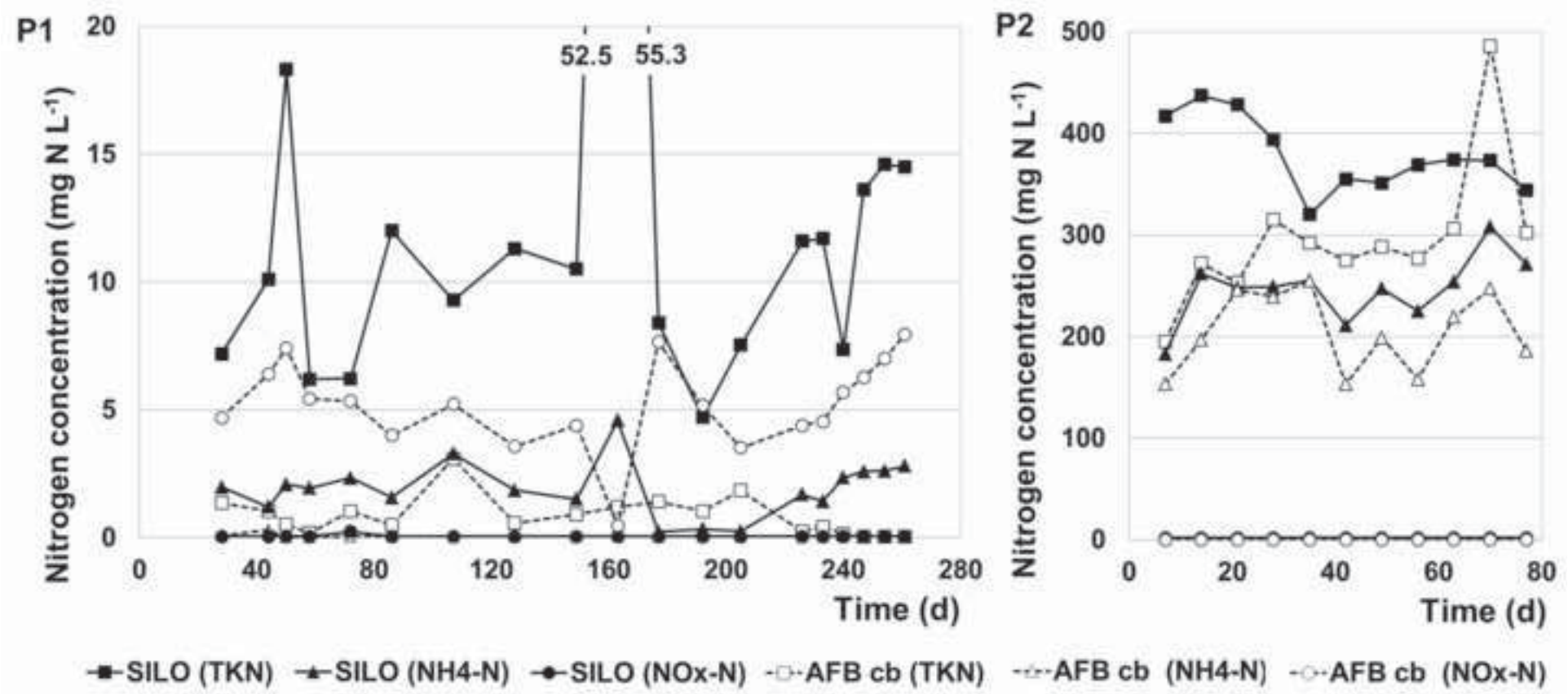
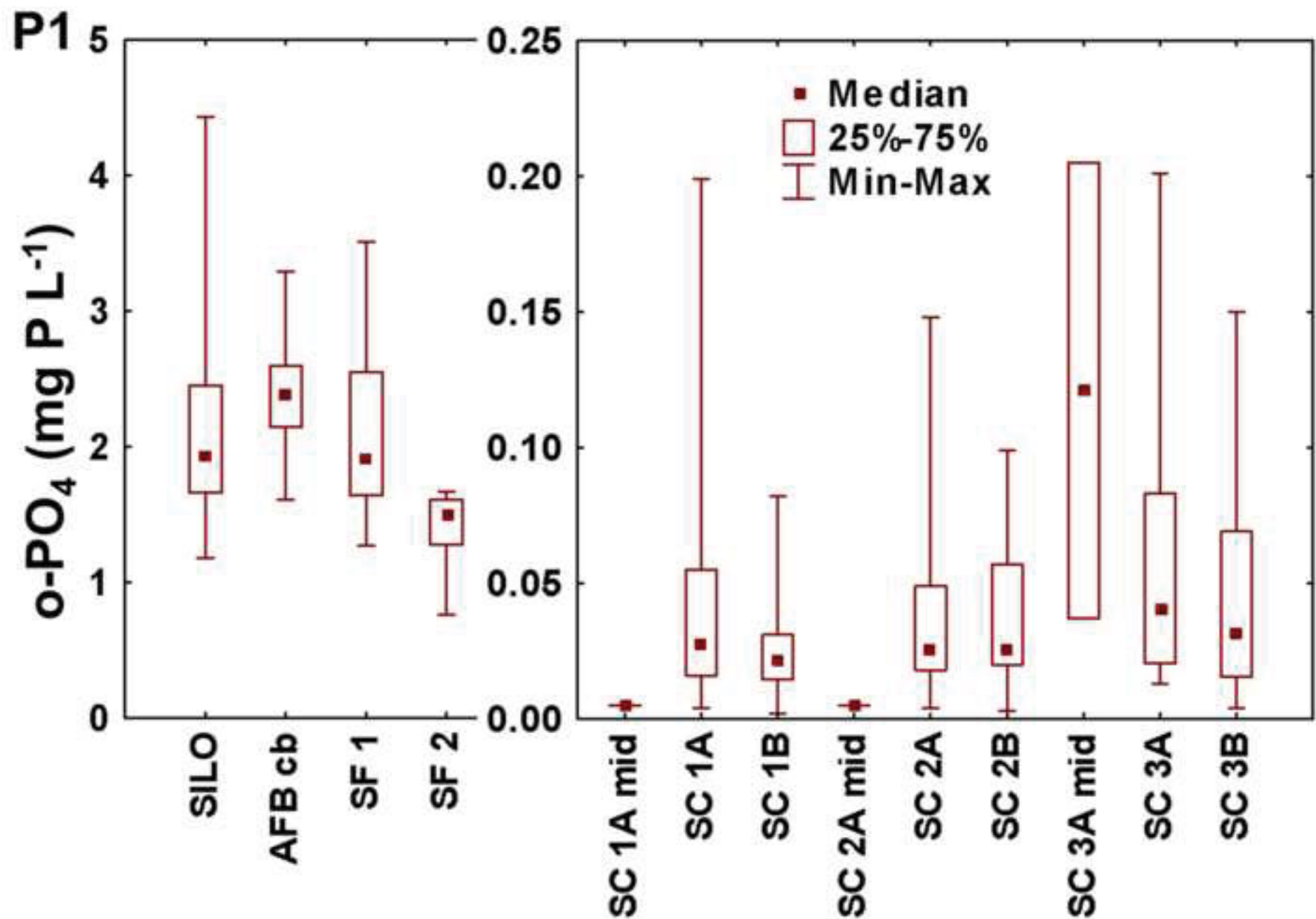


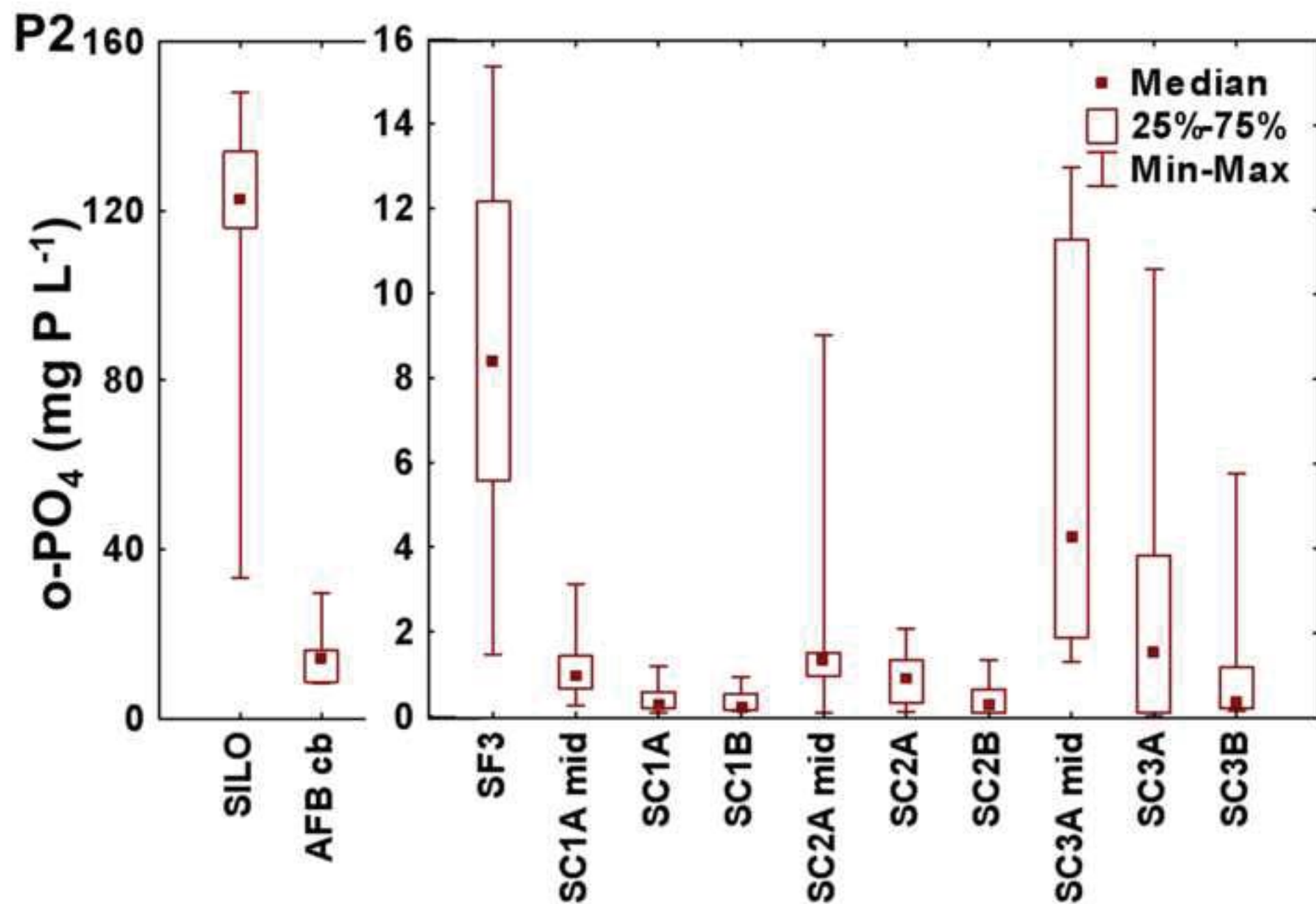


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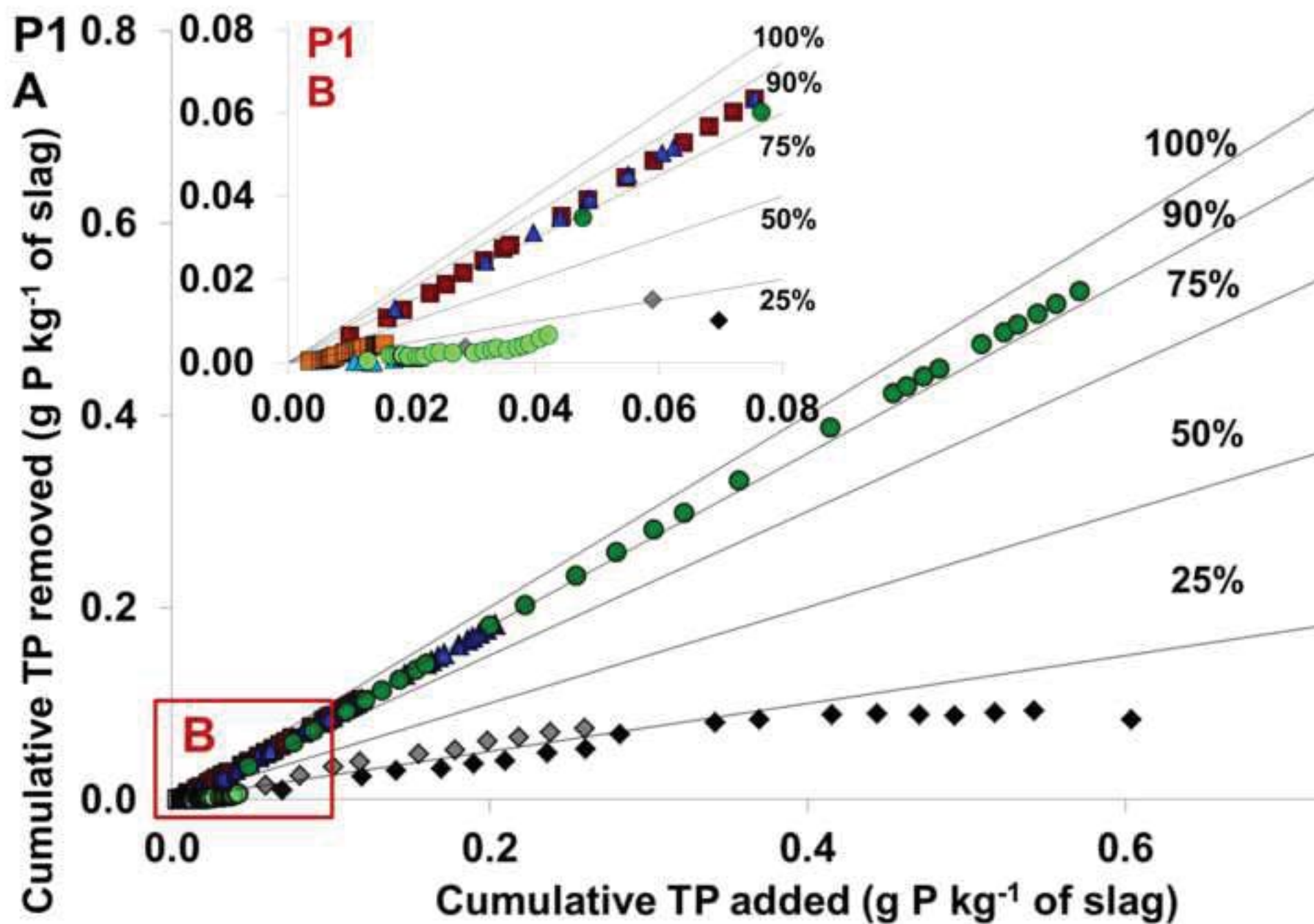
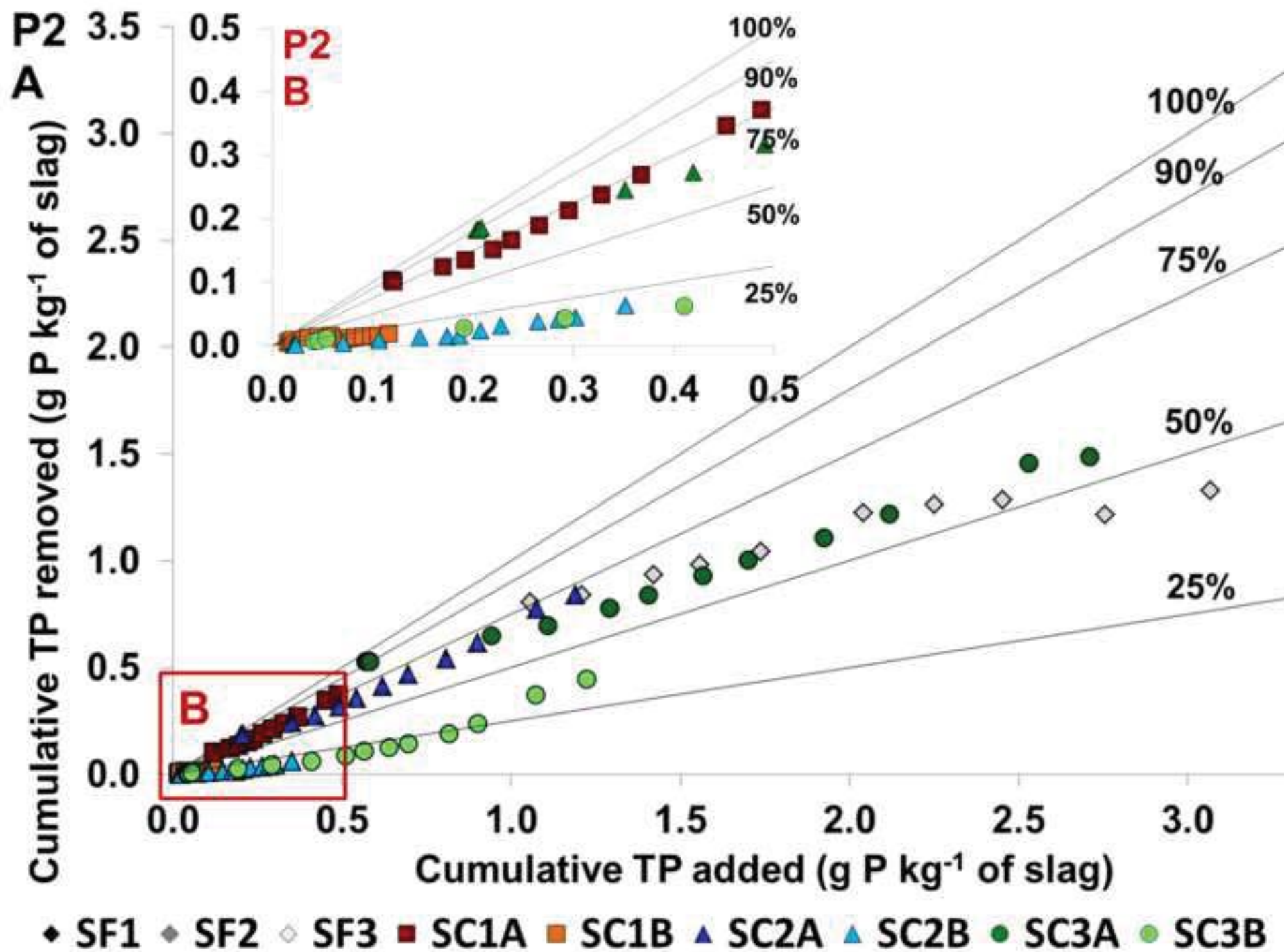
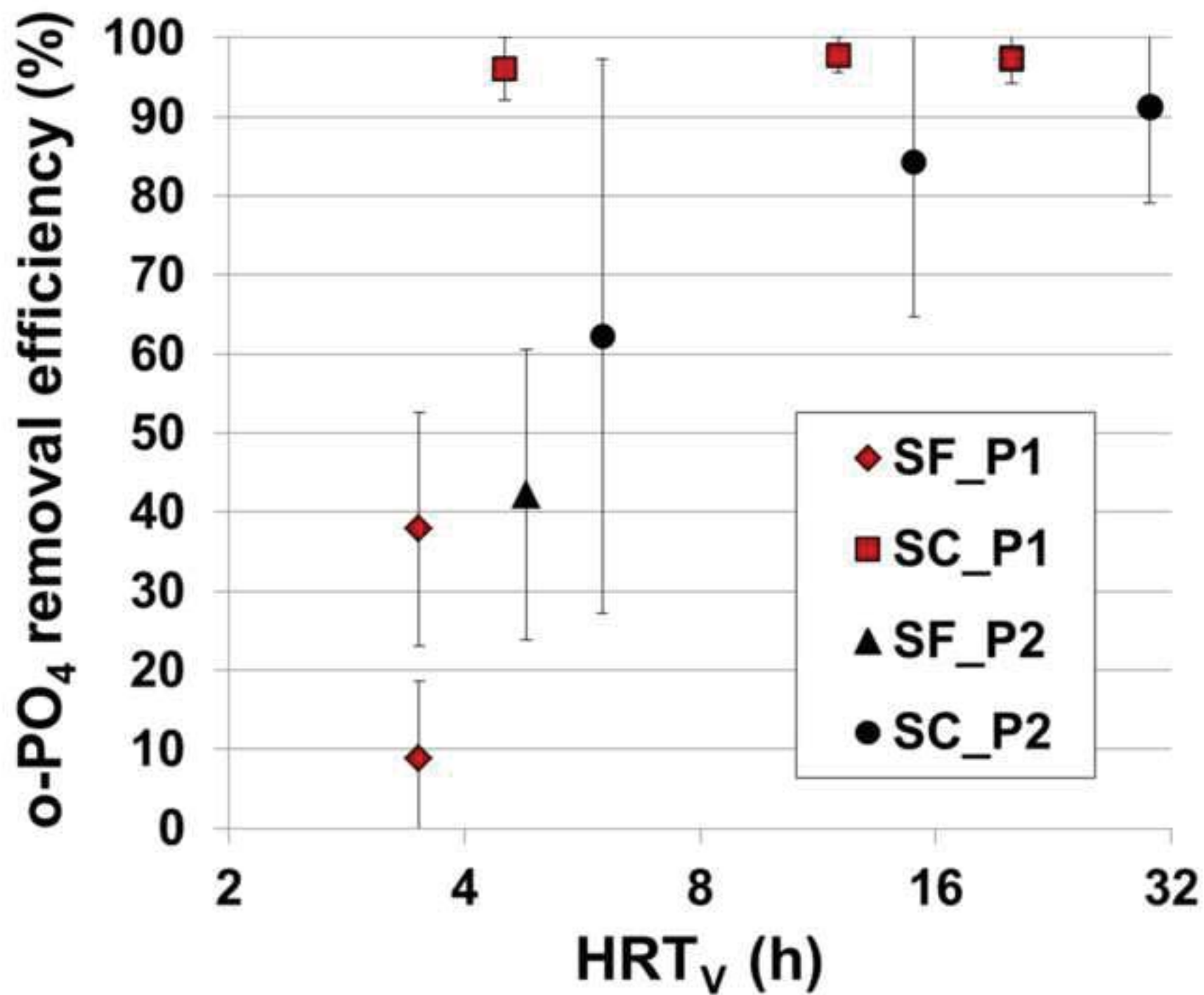


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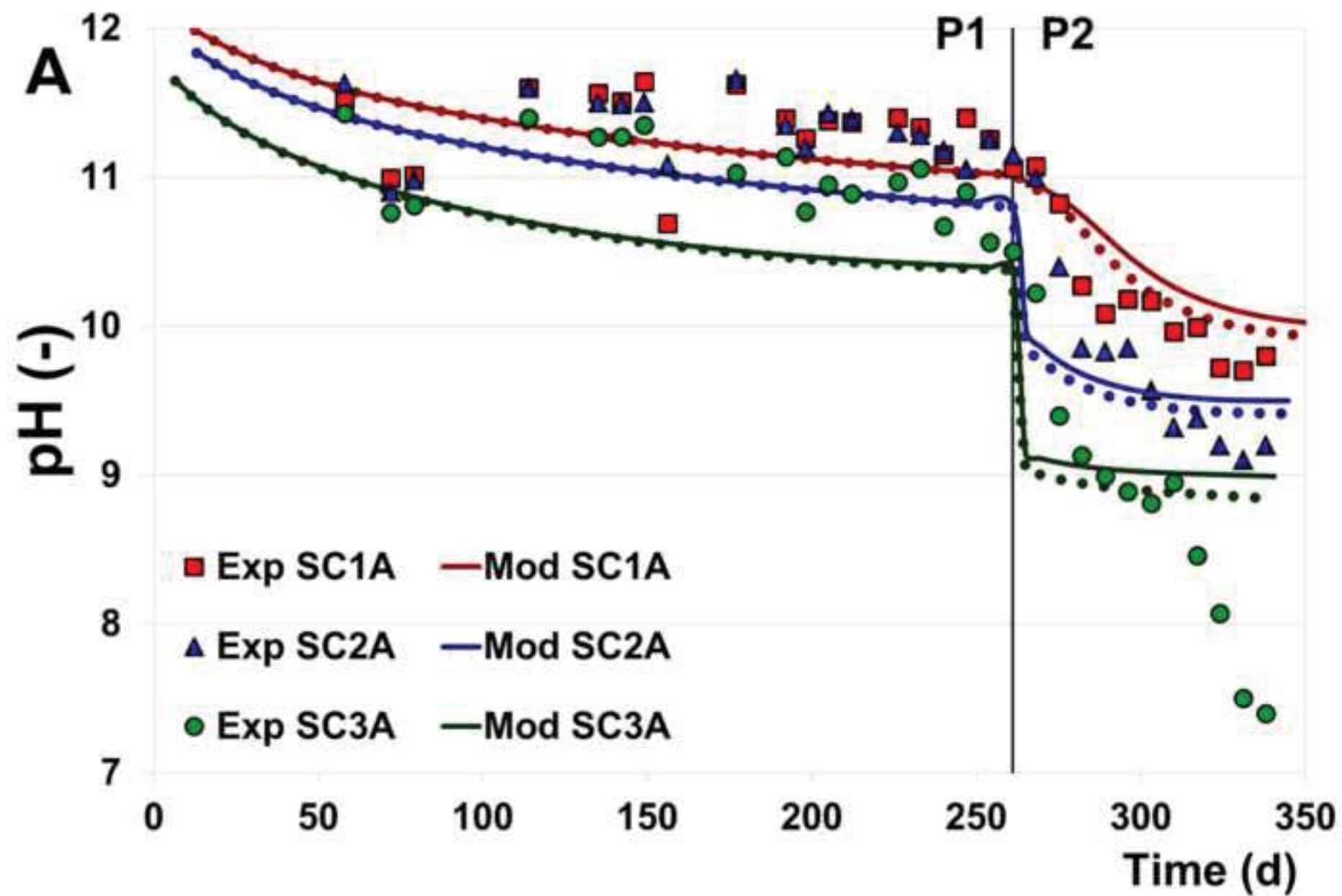
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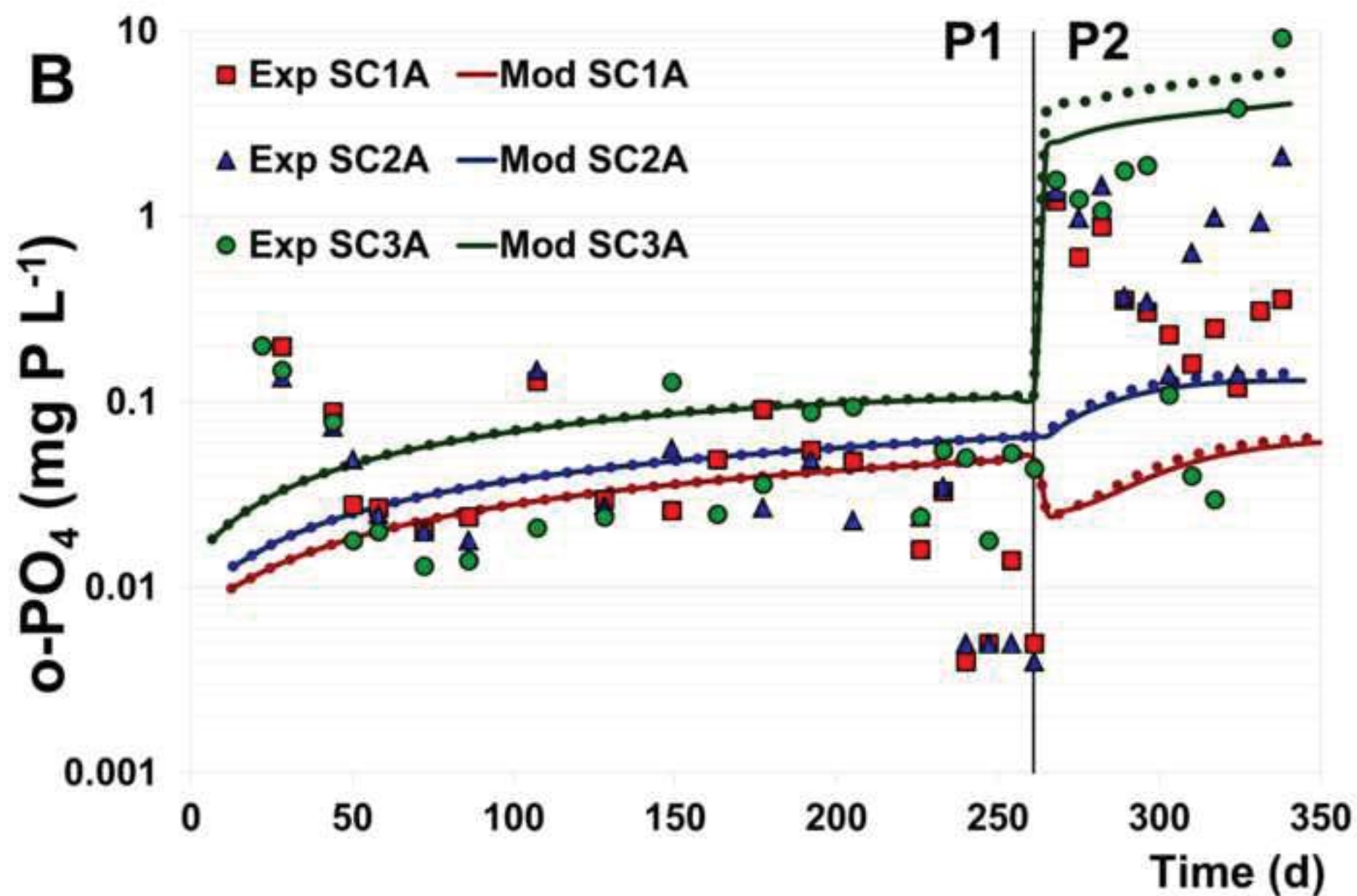




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## Koiv et al., TABLES

**Table 1.** Summary of design parameters of the experimental filter units in Phase 1 (P1) and Phase 2 (P2). Abbreviations: AFB1 and AFB2 – two parallel aerated filter beds; SF – sacrificial slag filter; SC1, SC2, SC3 – three parallel dual-stage steel slag columns.

Design parameters	Units	AFB1, AFB2	SF	SC1, SC2, SC3
Water flow conditions		saturated	saturated	saturated
		downflow	upflow	upflow
Filter size				
(diameter × height	m×m or	*1.0×1.0×1.0	0.45×0.8	0.3×1.3
or *length × width × height)	*m×m×m	(each AFB)		(each stage)
Water level in filter units	m	1.0	0.8	1.3
Volume of filter material	m <sup>3</sup> filter <sup>-1</sup> (*or stage <sup>-1</sup> )	0.90	0.13	*0.095
Filter material		gravel	EAF steel slag	EAF steel slag
Particle size of material	mm	10-25	P1 SF1 = 20-40	
			P2 SF2 = 10-30	5-10
			P2 SF3 = 10-30	
Density of material	kg L <sup>-1</sup>	2.6	3.6	3.6
Porosity of filter (estimated)	%	38	45	40
Initial void volume	m <sup>3</sup>	0.36	0.052	0.038
Void hydraulic retention time (HRT <sub>v</sub> )	h	P1 = 48	P1 = 3.5	P1: 20, 12, 4.5
		P2 = 65	P2 = 4.8	P2: 30, 15, 6.0 (per stage)
Organic loading rate (OLR)	kg BOD <sub>5</sub> m <sup>-3</sup> d <sup>-1</sup>	P1 = 0.015 P2 = 0.50	—	—
Air flow direction		counter-	—	—



Design parameters	Units	AFB1, AFB2	SF	SC1, SC2, SC3
current				

**Table 2.** Composition of simulated influent solutions of the slag columns (AFBs effluent feeding the SF and SF effluent feeding the SCs).

Phase	pH	Ca <sup>2+</sup>	TIC	o-PO <sub>4</sub>	NH <sub>4</sub>	Alkalinity
	-	mg L <sup>-1</sup>	mg C L <sup>-1</sup>	mg P L <sup>-1</sup>	mg N L <sup>-1</sup>	mg CaCO <sub>3</sub> L <sup>-1</sup>
AFB P1	7.19	24.6	13.4	2.42	-	51.6
SF P1	8.27	28.1	13.4	2.42	-	60.3
AFB P2	7.39	23.4	159.8	13.6	210	645.3
SF P2	8.07	30.7	145.2	13.6	210	663.4

**Table 3.** Preliminary full-scale design parameters for the treatment of low (P1) and high strength (P2) supernatant of a freshwater fish farm sludge settling tank, consisting of a) an aerated filter bed (AFB) and b) a reactive slag filter.

Wastewater type		P1 (low strength)	P2 (high strength)
<b>Aerated filter bed</b>			
<b>1) AFB influent data (see Supplementary Table S2)</b>			
<b>2) Objectives</b>			
TKN removal efficiency	%	90	10
BOD removal efficiency	%	80	50
<b>3) AFB design criteria (results from trickling filter design, Metcalf and Eddy et al., 2014)</b>			
Type of AFB		low-rate	high-rate (with recirculation)
Specific surface area in the reactor	m <sup>2</sup> m <sup>-3</sup>	60	60
Hydraulic loading rate	m d <sup>-1</sup>	3.6	1.04
Organic loading rate	kg BOD <sub>5</sub> m <sup>-3</sup> d <sup>-1</sup>	0.13	1.57
Specific TKN removal rate per rock area	g m <sup>-2</sup> d <sup>-1</sup>	0.40	0.33
<b>4) Design values</b>			
Filter volume (empty bed)	m <sup>3</sup>	28	19
Filter depth	m	2	2

Filter area	m <sup>2</sup>	13.9	9.6
Particle size of material	mm	10-20	10-20
Aeration rate	m <sup>3</sup> min <sup>-1</sup>	0.83	2.92
<b>Slag filter</b>			
<b>1) Slag filter influent data (see Supplementary Table S2)</b>			
<b>2) Objectives</b>			
P removal efficiency	%	80	80
<b>3) Slag filter design criteria (results from numerical simulations using the P-Hydroslag model)</b>			
Void hydraulic retention time	h	8	30
Longevity	pore volumes	5400	315
	years	5	1
P retention capacity	g P kg <sup>-1</sup> slag	4.5	1.7
<b>4) Design values</b>			
Filter volume (empty bed)	m <sup>3</sup>	42	31
Filter depth	m	2	2
Filter area	m <sup>2</sup>	21	15.5
Particle size of material	mm	5-10	5-10

## Supplementary Material

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